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(A) A fluid gel obtainable by a process which comprises subjecting a solution comprising a mixture of xanthan and one or more non-gelling polysaccharides selected from galactomannans or glucomannans (e.g. konjac mannan or locust bean gum) in a solvent to shear while cooling the solution from a temperature above the gelation temperature of the mixture, and recovering the resulting continuous gel. The gel is useful in cosmetic and pharmaceutical compositions where it may, for instance, be generated in situ.

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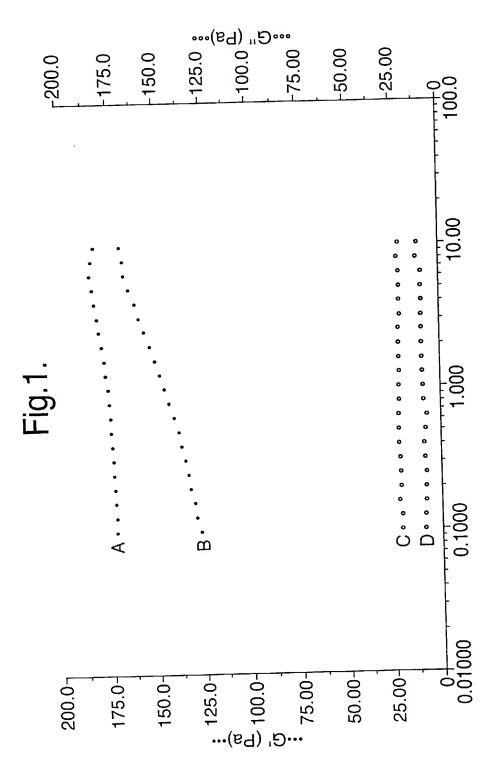
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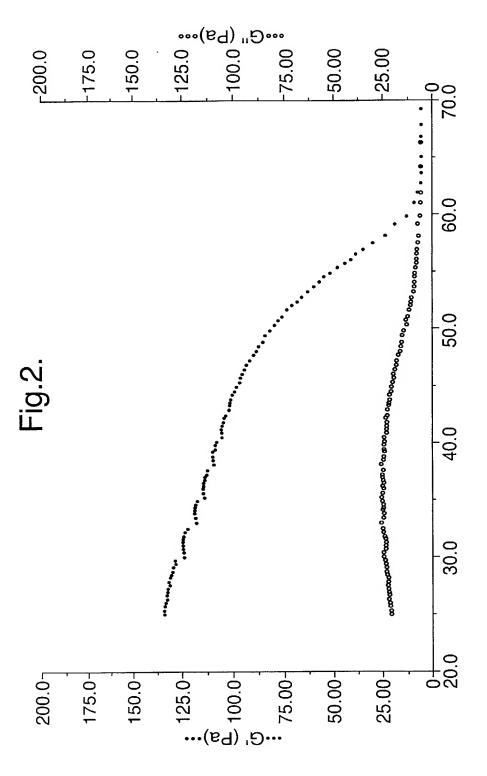
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- (54) Abstract Title New gel
- (57) A fluid gel obtainable by a process which comprises subjecting a solution comprising a mixture of xanthan and one or more non-gelling polysaccharides selected from galactomannans or glucomannans (e.g. konjac mannan or locust bean gum) in a solvent to shear while cooling the solution from a temperature above the gelation temperature of the mixture, and recovering the resulting continuous gel. The gel is useful in cosmetic and pharmaceutical compositions where it may, for instance, be generated in situ.





#### **NEW GEL**

The present invention relates to fluid gels of xanthan and non-gelling polysaccharides and processes for producing these fluid gels. The invention also relates to compositions comprising the fluid gels and uses of the gels and of the compositions.

There is a growing desire in the cosmetics industry to move away from the use of synthetic materials in gels and to use natural polymers such as polysaccharides instead. However, the gels formed from such polysaccharides tend to be rigid and consequently of limited use in cosmetic applications.

The present invention provides a fluid gel obtainable by a process which comprises subjecting a solution comprising a mixture of xanthan and one or more non-gelling polysaccharides selected from galactomannans and glucomannans in a solvent to shear while cooling the solution from a temperature above the gelation temperature of the mixture, and recovering the resulting continuous gel.

The gels of the present invention comprise xanthan and one or more non-gelling polysaccharides chosen from galactomannans and glucomannans. Examples of non-gelling polysaccharides include konjac mannan, tara, locust bean and guar gums. In a preferred embodiment of the present invention the gel comprises a mixture of xanthan and locust bean gum (lbg) or konjac mannan (km). In a particularly preferred embodiment the gel comprises a mixture of xanthan and konjac mannan.

Modified xanthan or a modified non-gelling polysaccharide may be used in the present invention. Modification may be, for example, by depolymerisation, chemical action or enzymatic action. Examples of modified products are depolymerised locust bean gum and enzyme modified guar gum.

Xanthan is an anionic bacterial polysaccharide whose commercial source is the bacterium *Xanthomonas campestris*. Xanthan is produced commercially by aerobic fermentation of this bacterium in batch culture. Precipitation with alcohol enables xanthan to be isolated from the culture broth. On removing the bacterial cells prior to alcohol precipitation a clarified product can be produced. Xanthan gum

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typically takes the form of an off-white powder with a moisture content of approximately 11% and an ash content of 6-9%. The viscosity of a xanthan solution is relatively insensitive to pH.

The viscosity of polysaccharide solutions normally decreases on addition of an electrolyte. However, for xanthan solutions of concentrations greater than 0.15% the electrolyte has the opposite effect and increases the viscosity. This is due to the electrolyte promoting helix formation. It is reported that maximum viscosity is obtained at a concentration of 0.02-0.07% sodium chloride. Addition of salt above this concentration has little or no effect on viscosity or stability. Addition of di- or trivalent cations also enhances the thixotropy of xanthan solutions.

The viscosity of aqueous xanthan solutions over a broad temperature range is almost constant. It has been shown that in the presence of salt, i.e. 0.1% concentration sodium chloride, the viscosity of xanthan solutions is practically unaffected by temperatures ranging from 4 to 93°C. This is contrary to most other polysaccharide solutions for which, on heating, the viscosity decreases.

Locust bean gum (lbg) is the refined endosperm of the seed of the carob tree known botanically as *Ceretonia siliqua*. The endosperm is separated from the husk and the germ of the seed by the process known as oven dehulling. This involves the seeds being heated to high temperatures for approximately 45 seconds and then being passed through a series of roller mills which enable the husk and germ to be removed simultaneously. It is important that complete removal of the husk is achieved during endosperm separation, otherwise specks may be observed in the gum which may cause loss in viscosity over time in prepared gum solutions.

Konjac mannan (km) is a linear polysaccharide derived from the konjac tuber of the *amorphophallus* species found in east Asia, from which a glucomannan flour may be obtained after subsequent drying and milling of the tuber. The polysaccharide has been used for many years as a food component in India, Pakistan and Japan. Konjac mannan is water soluble and produces highly viscous solutions which exhibit pseudoplastic flow.

The xanthan and non-gelling polysaccharides may be present in the fluid gel of the present invention in equal or unequal amounts. Where the gel comprises

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xanthan and one non-gelling polysaccharide these two components are typically present in a weight ratio of from 1:10 to 10:1, preferably 9:1 to 1:1.

The gels of the present invention are fluid. Depending on the concentration of the solution and the choice of non-gelling polysaccharides, the gels range in viscosity from pourable to spreadable gels. Gels formed from solutions of a lower concentration are generally pourable and gels formed from solutions of a higher concentration are generally spreadable. Pourable gels may typically be formed from solutions of 0.1% by weight of polysaccharide or lower.

In one embodiment the gels of the present invention are spreadable. The term "spreadable" means that the gel flows at low strain; for example, it can be spread easily by rubbing (possibly into the skin). When it flows the gel has a very smooth consistency. The gel flow proceeds smoothly at low strain and is different from the fracturing observed when a rigid gel is subjected to strain. The fluid (for instance spreadable) gels of the present invention, being formed by a process which includes a shearing step, are substantially less rigid than non-sheared gels. The gels are also noted for their good 'skin feel'. When applied to the skin, the gels rub in well and do not leave the skin feeling tight or sticky.

Gels are typically characterised by their rheological properties such as the storage (G') and loss (G") moduli. The (stronger and) more rigid a gel is, the higher the value of G'. The value of G" is generally lower than the value of G'.

G' and G" are measured in the linear viscoelastic region where stress is linearly proportional to strain.

The gels of the present invention are fluid and have a lower value of G' than gels formed under non-sheared conditions. It is thought that the shear applied as gelation is occurring serves to disrupt the gel structure, thereby inhibiting the formation of a complete interpenetrating three dimensional network and thus forming slightly weaker and softer gels.

The present invention also provides a process for producing a fluid gel, which process comprises subjecting a solution comprising a mixture of xanthan and one or more non-gelling polysaccharides chosen from galactomannans and glucomannans in a solvent to shear while cooling the solution from a temperature above the gelation

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temperature of the mixture, and recovering the resulting continuous gel.

It is an essential part of the process that the gel is subjected to shear as it cools through the gelation temperature. By subjecting to shear is to be understood subjecting to any suitable form of agitation which results in a fluid gel. For example, the solution may be sheared, stirred, shaken, disrupted, homogenised or agitated in any other way such as using ultrasound. Shearing may be conducted at a constant rate, i.e. controlled shearing, for instance in a Rheometer. Shearing may also be conducted at a non-constant rate, i.e. non-controlled shearing, for instance with a mechanical stirrer. Any suitable rate of shearing may be used.

Applying shear to the xanthan-lbg and xanthan-km systems while gelation occurs leads to the formation of soft, fluid continuous gels. These are different from other sheared gels, such as those described in EP-A-0 355 908 and EP-A-0 432 835 which are described as being particulate and do not tend to be fluid and continuous. The rheological profiles of the sheared gels of the present invention thus differ from those of non-sheared gels.

Typically, the shearing takes place while the solution cools from above the gelation temperature of the mixture of constituent polysaccharides, typically to room temperature. Normally the cooling takes place naturally, that is the gel cools gradually to room temperature. However either natural cooling or quench cooling may be used. Any manner of cooling which results in a fluid gel may be used. The solution may also be alternately quench cooled and naturally cooled when appropriate.

The shearing and cooling are typically simultaneous. Shearing may be conducted throughout the entire cooling process, or only during cooling through the gelation temperature. Shearing and cooling may take place contemporaneously or sequentially, for instance as alternating steps. For example, the solution may be sheared while being held at a constant temperature and then allowed to cool, followed by further shearing at a lower constant temperature and subsequent cooling. However, it is essential that shearing takes place as the solution gels, that is as the solution cools through the gelation temperature.

The xanthan and non-gelling polysaccharide solution generally comprises

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from 0.01 to 5% by weight of xanthan and non-gelling polysaccharide, typically from 0.05 to 2% and often 1%. A typical solution is produced by dissolving dried xanthan and non-gelling polysaccharide in distilled water such that the final polymer concentration is from 0.05 to 5%. Particularly preferred are solutions of 0.1 - 1% concentration. The preferred concentration for any particular combination of xanthan and polysaccharide depends on the non-gelling polysaccharide(s) chosen.

The solutions of xanthan and other non-gelling polysaccharides used in the present invention are typically aqueous solutions. Generally the dried xanthan and non-gelling polysaccharide(s) are dissolved in distilled water and then heated. However, solutions may be formed from other solvents or solvent mixtures. Examples of other solvents which are suitable include alcohols such as methanol, ethanol, isopropanol, cetyl alcohol, stearyl alcohol and glycols, ketones such as acetone and benzophenone and acetates. These solvents may be used alone, as mixtures with each other or mixed with water.

The solution is typically heated to above the gelation temperature of the mixture of xanthan and said one or more non-gelling polysaccharides. The solution is therefore typically heated to above 50°C and generally to above 70°C. However, the solution may be heated to above 90°C.

The solution is then allowed to cool to below the said gelation temperature. The gelation temperature may be defined as the temperature at which G' increases markedly. Typically the solution is allowed to cool to room temperature which is from 15 to 30°C, more preferably from 18 to 24°C.

The viscosity of the resulting gel is partly dependent on the temperature to which the starting xanthan and non-gelling polysaccharide solution is heated prior to cooling and shearing. For instance, the viscosity of a xanthan-konjac mannan gel which results from heating the starting solution to 90°C is generally higher than the viscosity of a corresponding gel which results from heating the starting solution to 70°C or 50°C. The properties of a gel of the invention may thus be changed by appropriate modification of the temperature to which the starting polysaccharide solution is heated. However, other factors such as the addition of electrolyte can also affect the properties of the gel.

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The xanthan and polysaccharide(s) are typically mixed as dry powders and then dissolved in solvents. However, making each constituent component of the gel of the invention as a separate solution and mixing the said solutions is also an embodiment of the present invention. The solutions may be mixed before or after heating to above the gelation temperature. For example the solutions may be mixed before heating, or after heating but before cooling to below the gelation temperature. The two (or more) solutions must come into contact before gelation occurs.

The gels of the present invention may be combined with other ingredients to form compositions, for instance cosmetic or pharmaceutical compositions for application to the skin. Examples of such compositions include creams, gels and lotions. They may also be used in toiletries including shampoos, deodorants, antiperspirants and toothpaste, in pharmaceutical compositions for application in the buccal cavity or to skin, in foods for animals or humans, for example confectionery, and in other materials such as agrochemicals, paints and inks. The compositions typically comprise a fluid gel of the present invention and a physiologically tolerable carrier, adjuvant or diluent. The composition may also comprise a pigment, a pharmaceutically active compound, a sunscreen active compound, a silicone or derivative thereof, an inorganic salt, a further solvent or a perfume.

Suitable ingredients for use in compositions comprising a gel of the present invention include 1,3-butylene glycol, acetaminophen, acetylated lanolin alcohol, acrylates copolymer, alpha olefin sulfonate, alumina, ammuonium lauryl sulfate, butylated hydroxytoluene, caprylic/capric tryiglcerides, carbomer, carbomer 940, carbomer 941, carboxymethyl cellulose, cellulose gum, centrimonium chloride, ceresin, cetearyl alcohol, cetearyl isononanoate, cetearyl octanoate, cetyl acetate, cetyl alcohol, cetyl dimethicone copolyol, cetyl trimethyl ammonium chloride, cholesterol, citric acid, cocamide DEA, cocamide MEA, cocamidopropyl betaine, cocoamphocarboxypropionate, coconut oil diethanolamine condensate, dicaprylyl maleate, diethanolamine, dimethicone, dipropylene glycol, disodium pareth-3 sulfosuccinate, ethanol, ethylene glycol monostearate, fatty acid ester, glycerin, glycerol monostearate (and) polyoxyethylene, glyceryl monostearate, glyceryl oleate, glyceryl stearate, glycol disterate, hexylene glycol, hydrated silica, hydroxyethyl

cellulose, hydroxypropyl guar hydroxypropyltrimonium chloride, isopropyl palmitate, kaolin, lactic acid, laneth-10 acetate, laneth-16, lanolin, lanolin alcohol, lanolin oil, lauramide DEA, laureth-3, lauric acid, lauric fatty acids, lauroamphodiacetate, lauroyl sarcosine, magnesium aluminium silicate, magesium stearate, magnesium sulphate, methyl gluceth-10 ethoxylated, methyl gluceth-20. mica, microcrystalline cellulose, mineral oil, oleamide MIPA, oleic acid, oleth-3 phosphate, oleyl alcohol, paraffinium liquidum, PEG-18 glyceryl oleate/cocoate, PEG-20 stearate, PEG-40 castor oil, PEG-40 hydrogenated castor oil, PEG-6 Cocamide, PEG-7 glyceryl cocoate, petrolatum, pigments, polyethylene, polyoxypropylene 15 stearyl ether, polyquaternium-7, polysorbate 20, polysorbate 60. polysorbate 80. potassium acesulfame, potassium hydroxide, propylene glycol, silica, sodium aluminium chlorhydroxylactate, sodium chloride, sodium citrate, sodium fluoride, sodium hydroxide, sodium laureth sulfate, sodium lauroyl sarcosinate, sodium lauryl sulfate, sodium monofluorophosphate, sodium N-methyl-N-myristoyl-taurate, sodium saccharin, sorbitan isostearate, sorbitan monopalmitate fatty acid ester, sorbitol, stearamidopropyl dimethylamine, stearate fatty acid ester, steareth-2, steareth-21, stearic acid, stearyl alcohol, stearyl alcohol (and) ceteareth-20 condensate, synthetic beeswax, talc, tetrasodium EDTA, theobromo cacao, titanium dioxide, titanium dioxide MT100T, triclosan, triethanolamine, xanthan gum, zinc oxide (Nanox) and zinc pyrithione (dispersion).

The further solvent may be an organic solvent with a low boiling point, for example, an ether which could not be added until after the solution has cooled. However, any suitable organic solvent may be added to the composition after cooling.

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In a further embodiment of the invention, other components of the final composition may be added to the solution before shearing takes place. These may be added at any suitable temperature. For example, certain solvents and perfumes are typically added at low temperatures in order not to vaporise, whereas inorganic salts may be added at any temperature.

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In one embodiment of the present invention the gel of the present invention is formed in situ in the composition. In this embodiment the gel components are

typically provided in a dry form, e.g. as powder, and the composition is subjected to shear after the gel components have been added. Further components may be added after the shearing step has taken place. Accordingly, the invention further provides a cosmetic or pharmaceutical composition obtainable by a process which comprises:

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(a) forming an aqueous phase comprising one or more cosmetically or pharmaceutically acceptable carriers, adjuvants or diluents and a mixture of xanthan and one or more non-gelling polysaccharides selected from galactomannans and glucomannans;

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- (b) Subjecting the aqueous phase to shear while cooling it from a temperature above the gelation temperature of the said mixture; and
- (c) recovering the resulting cosmetic or pharmaceutical composition.

The invention also provides a process for producing a cosmetic or pharmaceutical composition which process comprises:

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(a) forming an aqueous phase comprising one or more cosmetically or pharmaceutically acceptable carriers, adjuvants or diluents and a mixture of xanthan and one or more non-gelling polysaccharides selected from galactomannans and glucomannans;

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(b) Subjecting the aqueous phase to shear while cooling it from a temperature above the gelation temperature of the said mixture; and

(c) recovering the resulting cosmetic or pharmaceutical composition.

A composition of the present invention may be formulated to be a topical, oral or mucosal contact gel or lotion.

The spreadable gels of the present invention are typically optically clear. The gels can therefore be used in many therapeutic and/or diagnostic techniques. For example, they are suitable for use in ultrasound or ECG tests.

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The invention is further described in the Examples which follow, with reference to the accompanying drawings, in which xanthan, locust bean gum and konjac mannan were all obtained from commercial sources. G' and G" were measured using a Carrimed CSL<sup>2</sup>500 controlled stress rheometer (TA Instruments, UK).

#### **Drawings**

Figure 1 shows a comparison of G' and G" (y axis) for sheared and non-sheared xanthan-konjac mannan gels of 0.5% concerntration at 25°C in water over a frequency range of 0.1 to 10 Hertz (Hz) (x axis). Plot A relates to G' for non-sheared gels, plot B to G' sheared gels, plot C to G" for sheared gels and plot D to G" for non-sheared gels.

Figure 2 shows the change in G' and G" (y axis) with temperature in °C (x axis) of a sheared xanthan-konjac mannan gel of 0.5% concentration in water.

# 10 Example 1

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Xanthan-locust bean gum controlled shear gels were prepared as follows. The individual polysaccharides were weighed and dispersed in distilled water separately. Each polysaccharide solution was heated to 90°C while stirring with a magnetic stirrer for 1 hour. The solution was removed from the heat and water evaporation was accounted for by the addition of hot distilled water. The xanthan and locust bean gum solutions were mixed at ~90°C in the ratios described below. The sample was transferred to the Carrimed Rheometer and cooled to room temperature while shearing at a constant rate (for example 0.3s<sup>-1</sup> or 550s<sup>-1</sup>).

Solutions were prepared at a 0.25% and 0.5% total polymer concentration and the gums were mixed at a ratio of 80:20, 50:50 and 20:80.

The variation of G' and G" as a function of frequency for 0.5% concentration xanthan-lbg gels (20:80 ratio) sheared at two different shear rates, i.e., 0.3s<sup>-1</sup> and 550s<sup>-1</sup> is shown in Table 1. G' was greater than G" over the frequency range studied for both samples and both are independent of frequency. G' and G" values are approximately the same for the samples sheared at 550s<sup>-1</sup> and at 0.3s<sup>-1</sup>. The effect of controlled shear on the frequency sweeps of 0.25% concentration xanthan-lbg gels (80:20 ratio) sheared at the two different constant shear rates, i.e., 300s<sup>-1</sup> and 550s<sup>-1</sup> (shown in Table 2) showed that G' and G" are similar for both shear rates and that G' is greater than G" over the frequency range.

xan-lbg (0.5% c	onc.) in wa	ter - 550 s <sup>-1</sup>	xan-lbg (0.5% c	onc.) in w	ater - 0.3 s <sup>-1</sup>
frequency (Hz)	G' (Pa)	G'' (Pa)	frequency (Hz)	G' (Pa)	G'' (Pa)
1.00	31.53	2.21	1.00	26.10	1.42
1.18	31.79	2.16	1.18	26.20	1.47
1.39	32.00	2.20	1.39	26.46	1.29
1.64	32.03	2.20	1.64	26.46	1.51
1.93	32.27	2.31	1.93	26.25	1.39
2.28	32.45	2.45	2.28	26.34	1.38
2.68	33.03	2.78	2.68	26.26	1.76
3.15	32.51	2.58	3.15	26.36	1.54
3.72	31.82	3.04	3.72	25.73	1.79
4.39	31.44	2.93	4.39	25,32	1.99

# Table 2

xan-lbg (0.25%	conc.) in w	ater - 550 s <sup>-1</sup>	xan-lbg (0.2	5% conc.) in	water - $300 s^{-1}$
frequency (Hz)	G' (Pa)	G'' (Pa)	frequency	G' (Pa)	G'' (Pa)
1.00	10.99	0.92	1.00	13.83	0.97
1.18	10.79	0.77	1.18	13.58	1.07
1.39	11.17	0.96	1.39	13.44	1.27
1.64	11.12	1.07	1.64	13.62	1.22
1.93	11.19	1.04	1.93	13.58	1.17
2.28	11.39	1.23	2.28	13.38	1.05
2.68	11.45	1.09	2.68	12.92	1.09
3.15	10.45	1.08	3.15	12.47	1.69

#### Example 2

A non-controlled shear xanthan-locust bean gum gel was prepared as follows, Xanthan and locust bean gum powder were mixed in a weight ratio of 1:1. The mixed powder was dispersed in distilled water to form a 0.5% total polymer concentration. The solution was heated to 90°C while stirring with a mechanical stirrer for 1 hour. The solution was removed from the heat and water evaporation was accounted for by the addition of hot distilled water. The sample was cooled to room temperature and sheared using an overhead stirrer. The mixtures were either cooled naturally with stirring to room temperature or quench cooled with stirring to about 20°C.

The variation of G' and G" with frequency at 25°C was compared for quench cooled and naturally cooled samples. The results are shown in Table 3. For both gels G' was greater than G" over the frequency range studied for the samples and both G' and G" were independent of frequency. The results are shown in Table 3 and indicate that G' and G" are slightly higher when cooled naturally.

Table 3

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xan-lbg (0.5% c	onc.) in w	vater -	xan-lbg (0.5%	conc.) in	water -		
quench cooled			cooled naturally				
frequency (Hz)	G' (Pa)	G'' (Pa)	frequency (Hz	) G' (Pa)	G'' (Pa)		
1.00	123.50	8.44	1.00	129.00	13.61		
1.18	121.30	8.10	1.18	132.50	12.25		
1.39	123.00	5.97	1.39	129.40	12.92		
1.64	117.60	8.36	1.64	130.10	13.92		
1.93	123.60	8.04	1.93	130.20	13.02		
2.28	122.10	7.64	2.28	129.50	12.84		
2.68	122.60	8.29	2.68	131.00	15.69		
3.15	121.30	6.46	3.15	133.40	14.04		
3.72	119.80	8.87	3.72	132.90	15.00		
4.39	118.40	9.48	4.39	131.50	15.20		
5.17	111.60	8.47	5.17	132.00	14.48		
6.10	111.50	6.05	6.10	131.80	18.26		
7.18	107.70	12.10	7.18	134.50	19.29		
8.48	102.70	9.14	8.48	137.70	19.47		
10.01	104.20	14.34	10.01	145.80	19.23		

#### Example 3

A xanthan-konjac mannan controlled shear gel was prepared following the procedure described in Example 1. The gums were mixed at a ratio of 1:1 and a 0.5% concentration solution was prepared. The shear rate used was 550s<sup>-1</sup>. A non-controlled shear xanthan-konjac mannan gel was prepared using the procedure of Example 2.

G' and G" were compared for the controlled and non-controlled shear

xanthan-konjac mannan gels at 25°C. The results are shown in Table 4. For both gels G' was greater than G" over the frequency range studied and both were independent of frequency. G' and G" were slightly less for the controlled shear gels than the non-controlled shear gels.

Gels were also prepared at 0.25% concentration and the effect of concentration on the frequency sweeps of xanthan-km controlled shear gels was studied. The results are shown in Table 5. For both concentrations G' was greater than G" over the whole frequency range and both were independent of frequency. G' and G" values were noticeably higher for the 0.5% concentration xanthan-km controlled shear gel than for the 0.25% concentration xanthan-km controlled gel.

Table 4

xan-km (0.5% c	onc.) in w	ater -	xan-km (0.5% conc.) in water -				
controlled shear	550 1/s		non-controlled shear				
frequency (Hz)	G' (Pa)	G'' (Pa)	frequency (Hz)	G' (Pa)	G'' (Pa)		
1.00	140.90	5.24	1.00	164.80	12.85		
1.18	141.30	5.12	1.18	164.80	14.79		
1.39	141.40	5.02	1.39	163.90	14.73		
1.64	141.90	4.95	1.64	163.40	16.34		
1.93	142.50	5.06	1.93	160.90	18.65		
2.28	142.60	5.03	2.28	162.90	18.43		
2.68	142.90	5.26	2.68	164.60	24.26		
3.15	143.10	5.19	3.15	160.70	22.49		
3.72	143.40	5.32	3.72	159.90	22.19		
4.39	143.40	5.44	4.39	159.60	21.51		
5.17	143.10	5.76	5.17	155.30	23.79		
6.10	139.60	7.20	6.10	155.40	21.48		
7.18	137.40	7.96	7.18	154.90	20.67		
8.48	136.80	7.88	8.48	149.20	22.62		
10.01	136.50	7.99	10.01	148.80	25.46		

Table 5

xan-km controlled shear gel	xan-km controlled shear gel
at 0.25% conc.	at 0.5% conc.

frequency	G'	G''	frequency	G'	G''
Hz	Pa	Pa	Hz	Pa	Pa
1.00	75.86	3.24	1.00	140.90	5.24
1.18	75.79	2.92	1.18	141.30	5.12
1.39	75.86	3.06	1.39	141.40	5.02
1.64	75.85	3.04	1.64	141.90	4.95
1.93	75.64	3.06	1.93	142.50	5.06
2.28	75.70	2.95	2.28	142.60	5.03
2.68	75.46	2.74	2.68	142.90	5.26
3.15	75.60	2.93	3.15	143.10	5.19
3.72	75.03	3.25	3.72	143.40	5.32
4.39	73.56	4.49	4.39	143.40	5.44
5.17	72.80	3.53	5.17	143.10	5.76
6.10	72.83	3.69	6.10	139.60	7.20
7.18	72.07	3.44	7.18	137.40	7.96
8.48	72.73	3.78	8.48	136.80	7.88
10.01	73.21	4.59	10.01	136.50	7.99

Xanthan-lbg gels were prepared at 0.5% concentration of 1:1 xanthan: locust bean gum in water, 0.04 NaCl and 0.15 NaCl by the procedure described in Example 2.

The variation of G' and G" with frequency at 25°C was compared for gels in water, 0.04 NaCl and 0.15 NaCl. The results are shown in Table 6. For all solutions G' was greater than G" over the whole frequency range and G' was independent of frequency for all three mixtures. The values obtained for G' and G" were higher in water and decreased in the order water > 0.04M NaCl> 0.15M NaCl. On cooling the onset of gelation corresponded to the sudden rise in G'. This occurred at about 57°C for all three systems (data not shown).

#### Table 6

xan-lbg sh	eared	gel	xan-lbg sh	xan-lbg sheared gel (0.5%			xan-lbg sheared gel (0.5%		
(0.5% con	c.) in	water	conc.) in 0.	04M	NaCl	conc.) in 0.	15M I	NaCl	
frequency	G'	G''	frequency	G'	G''	frequency	G'	<b>G</b> ''	
Hz	Pa	Pa	Hz	Pa	Pa	Hz	Pa	Pa	

	0.10	120.00	11.08	0.10	35.01	4.75	0.10	17.97	2.56
	0.13	122.20	10.68	0.13	35.51	4.77	0.13	18.41	2.45
	0.16	124.00	10.52	0.16	36.28	5.06	0.16	18.78	2.72
	0.20	125.70	10.32	0.20	36.92	5.20	0.20	19.40	2.85
	0.25	127.20	10.45	0.25	37.59	5.56	0.25	19.83	2.94
	0.32	128.60	10.29	0.32	38.23	5.61	0.32	20.37	2.93
	0.40	130.20	10.47	0.40	39.09	5.94	0.40	20.58	3.47
	0.50	131.40	10.46	0.50	39.74	6.02	0.50	20.91	3.32
ĺ	0.63	132.90	10.51	0.63	40.75	6.20	0.63	21.42	3.48
	0.79	134.40	10.87	0.79	41.45	6.57	0.79	21.91	3.73
	1.00	135.70	11.09	1.00	42.42	7.12	1.00	22.43	3.76
	1.26	137.10	11.42	1.26	43.22	7.41	1.26	23.04	4.25
	1.58	138.70	11.83	1.58	44.25	8.01	1.58	23.58	4.62
	2.00	140.50	12.31	2.00	45.46	8.44	2.00	23.82	4.85
	2.52	142.40	12.57	2.52	46.46	9.04	2.52	24.41	5.22
	3.15	143.90	13.07	3.15	47.78	9.72	3.15	24.75	5.64
	3.99	145.70	13.51	3.99	48.54	10.47	3.99	24.67	6.13
	5.00	147.70	14.25	5.00	49.12	11.29	5.00	24.64	6.94

To assess the effect of alcohol. The variation of G' and G" as a function of frequency for 0.5% concentration xanthan-lbg sheared gels in water and 20% methanol was studied. The results are shown in Table 7. G' was greater than G" over the entire frequency range and neither was dependent on frequency. The values obtained for the storage and loss moduli were similar for both systems.

Table 7

xan-lbg she	- `	0.5%		eared gel (0.5) % methano		
frequency	G'	G''	frequency	G'	G"	
Hz	Pa	Pa	Hz	Pa	Pa	
0.10	120.00	11.08	0.10	127.60	5.44	
0.13	122.20	10.68	0.13	127.80	6.16	
0.16	124.00	10.52	0.16	128.90	7.29	
0.20	125.70	10.32	0.20	129.50	5.32	
0.25	127.20	10.45	0.25	131.00	5.94	

0.32	128.60	10.29	0.32	132.10	5.94
0.40	130.20	10.47	0.40	134.00	9.62
0.50	131.40	10.46	0.50	133.70	6.92
0.63	132.90	10.51	0.63	135.30	6.63
0,79	134.40	10.87	0.79	135.90	6.84
1.00	135.70	11.09	1.00	138.20	7.30
1.26	137.10	11.42	1.26	138.10	8.99
1.58	138.70	11.83	1.58	139.10	8.82
2.00	140.50	12.31	2.00	141.40	8.88
2.52	142.40	12.57	2.52	142.20	9.48
3.15	143.90	13.07	3.15	142.40	10.96
3.99	145.70	13.51	3.99	143.30	11.73
5.00	147.70	14.25	5.00	143.70	11.82
6.29	149.50	15.10	6.29	142.10	12.02
7.93	149.30	17.02	7.93	139.50	12.99
10.01	150.80	17.40	10.01	137.90	13.83

Xanthan-lbg mixtures (0.5%, 50:50) were prepared and adjusted to pH 2 and pH 10 while hot (~70°C). The variation of G' and G" with frequency was compared for gels at pH 2, pH 5.5 and pH 10. The results are shown in Table 8. G' was greater than G" over the frequency range studied and both were independent of frequency for pH 5.5 and 10. G' and G" values were only slightly higher at pH 5.5 than in the sample adjusted to pH 10. However, for the xanthan-lbg mixture adjusted to pH 2, G" was greater than G' over the whole frequency range and both showed a strong frequency dependence (Table 8). G' and G" values at pH 2 were also lower than G' and G" values for mixtures at pH 10 or at pH 5.5.

Table 8

xan-lbg sh	eared g	el	xan-lbg sh	eared g	el	xan-lbg sheared gel		
(0.5% con-	c.) pH 5	.5	(0.5% con	c.) @ p	Н2	(0.5% conc	.) @ pH	[ 10
frequency	G'	G''	frequency	G'	G''	frequency	G'	G''
Hz	Pa	Pa	Hz	Pa	Pa	Hz	Pa	Pa
0.10	120.00	11.08	0.10	0.05	0.24	0.10	90.67	8.03
0.13	122.20	10.68	0.13	0.07	0.30	0.13	92.68	7.39

0.16	124.00	10.52	0.16	0.10	0.36	0.16	95.38	7.21
0.20	125.70	10.32	0.20	0.15	0.43	0.20	95.66	8.02
0.25	127.20	10.45	0.25	0.20	0.51	0.25	97.07	7.26
0.32	128.60	10.29	0.32	0.25	0.60	0.32	98.53	7.02
0.40	130.20	10.47	0.40	0.33	0.70	0.40	98.99	7.29
0.50	131.40	10.46	0.50	0.40	0.81	0.50	99.80	7.43
0.63	132.90	10.51	0.63	0.49	0.93	0.63	101.70	7.45
0.79	134.40	10.87	0.79	0.62	1.08	0.79	102.50	8.10
1.00	135.70	11.09	1.00	0.69	1.24	1.00	104.30	7.91
1.26	137.10	11.42	1.26	0.83	1.37	1.26	105.70	8.08
1.58	138.70	11.83	1.58	0.91	1.55	1.58	105.80	8.57
2.00	140.50	12.31	2.00	1.05	1.90	2.00	107.00	8.26
2.52	142.40	12.57	2.52	0.93	2.08	2.52	108.70	8.49
3.15	143.90	13.07				3.15	109.50	9.26
3.99	145.70	13.51				3.99	109.70	9.06
5.00	147.70	14.25				5.00	110.90	9.84
6.29	149.50	15.10				6.29	111.40	10.67
7.93	149.30	17.02				7.93	110.60	11.20
10.01	150.80	17.40				10.01	110.60	11.19

Xanthan-lbg mixtures (0.5%, 50:50) were prepared by heating the aqueous polysaccharide solutions to 90°C, 70°C and 50°C using the procedure outlined in example 2. The mixed solutions were then cooled naturally and sheared.

The variation of G' and G" with frequency for gels mixed at 90°C, 70°C and 50°C was compared. The results are shown in Table 9. For all three samples G' was greater than G" at 25°C over the entire frequency range and all were independent of frequency. From the three temperatures studied, it is seen that G' and G" increase when the mixture is subjected to higher temperatures.

Table 9

xan-lbg sheared gel		xan-lbg sheare	d gel	xan-lbg sh	xan-lbg sheared gel		
(0.5% conc.) in	water	(0.5% conc.) in water (0.5% conc.) in v		water			
90°C		70°C		50°C			
frequency G'	G''	frequency G'	<b>G</b> ''	frequency	G'	<b>G</b> ''	

Hz	Pa	Pa	Hz	Pa	Pa	Hz	Pa	Pa
0.10	120.00	11.08	0.10	86.52	9.96	0.10	75.02	6.38
0.13	122.20	10.68	0.13	88.49	9.53	0.13	75.66	5.57
0.16	124.00	10.52	0.16	90.33	9.12	0.16	76.05	5.77
0.20	125.70	10.32	0.20	91.86	9.02	0.20	76.47	5.26
0.25	127.20	10.45	0.25	92.93	8.91	0.25	76.83	5.32
0.32	128.60	10.29	0.32	94.40	9.31	0.32	77.49	5.70
0.40	130.20	10.47	0.40	95.33	9.07	0.40	78.41	4.95
0.50	131.40	10.46	0.50	96.47	9.33	0.50	78.86	5.43
0.63	132.90	10.51	0.63	97.55	9.31	0.63	79.16	5.08
0.79	134.40	10.87	0.79	98.76	9.98	0.79	79.68	5.14
1.00	135.70	11.09	1.00	99.97	10.22	1.00	80.48	5.39
1.26	137.10	11.42	1.26	101.20	10.50	1.26	80.70	4.97
1.58	138.70	11.83	1.58	102.60	10.94	1.58	81.32	5.37
2.00	140.50	12.31	2.00	103.90	11.32	2.00	81.65	5.60
2.52	142.40	12.57	2.52	105.50	11.71	2.52	82.33	5.54
3.15	143.90	13.07	3.15	107.20	12.05	3.15	82.93	6.13
3.99	145.70	13.51	3.99	108.90	12.81	3.99	83.26	6.27
5.00	147.70	14.25	5.00	109.70	13.64	5.00	83.57	6.70
6.29	149.50	15.10	6.29	110.80	15.38	6.29	83.36	6.53
7.93	149.30	17.02	7.93	111.50	15.44	7.93	83.15	7.17
10.01	150.80	17.40	10.01	113.10	15.86	10.01	83.62	7.72

Xanthan-konjac mannan gels were prepared at a ratio of 1:1 and stirred at 550rpm to study the effect of the rate of cooling.

The variation of G' and G" with frequency for xanthan-konjac mannan gels was compared for naturally cooled and quench cooled gels. The results are shown in Table 10. For both the quench cooled and naturally cooled samples G' was greater than G" over the frequency range studied and both were independent of frequency. However, G' and G" values obtained for the xanthan-km mixture cooled naturally to room temperature were higher than those obtained for the quench cooled mixture.

# Table 10

xan-km she	ared gel (	(0.5%	xan-km sh	eared go	el (0.5%		
conc.) in wa	ater coole	ď	conc.) in w	ater qu	ench		
naturally			cooled				
frequency	G'	<b>G</b> ''	frequency	G'	G''		
Hz	Pa	Pa	Hz	Pa	Pa		
1.00	164.80	12.85	1.00	78.11	6.41		
1.18	164.80	14.79	1.18	83.05	7.80		
1.39	163.90	14.73	1.39	81.28	5.31		
1.64	163.40	16.34	1.64	78.16	7.70		
1.93	160.90	18.65	1.93	79.16	7.54		
2.28	162.90	18.43	2.28	81.60	5.88		
2.68	164.60	24.26	2.68	80.80	6.28		
3.15	160.70	22.49	3.15	79.48	8.18		
3.72	159.90	22.19	3.72	74.65	6.55		
4.39	159.60	21.51	4.39	73.99	6.98		
5.17	155.30	23.79	5.17	73.89	8.76		
6.10	155.40	21.48	6.10	70.37	6.70		
7.18	154.90	20.67	7.18	66.47	6.03		
8.48	149.20	22.62	8.48	66.28	8.74		
10.01	148.80	25.46	10.01	64.07	9.00		

Xanthan-km sheared gels were prepared at 0.5% concentration in water, 0.04M NaCl and 0.15M NaCl. The variation of G' and G" with frequency was compared for the three samples. The results are shown in Table 11. Typical gel-like spectra (i.e. G' is always higher than G" and independent of frequency) were obtained for all three samples. G' and G" values are appreciably higher in the absence of electrolyte and decrease in the order no electrolyte> 0.04M NaCl> 0.15 NaCl.

The effect of zinc chloride on xanthan-km systems was also studied. The results are shown in Table 12. The variation of G' and G" as a function of frequency for xanthan-km sheared gels in water and 0.1M ZnCl<sub>2</sub> were compared. G' and G" values were significantly higher in water than in zinc chloride.

#### 35 Table 11

			xan-km sh	eared g	gel	xan-km sh	eared g	el
xan-km sh	eared gel	(0.5%	(0.5% con	c.) in 0.	04M	(0.5% con	c.) in 0.	15M
conc.) in w	ater		NaCl			NaCl		
frequency	G'	<b>G</b> ''	frequency	G'	<b>G</b> ''	frequency	G'	<b>G</b> ''
Hz	Pa	Pa	Hz	Pa	Pa	Hz	Pa	Pa
1.00	145.80	21.58	1.00	52.23	6.36	1.00	26.53	8.95
1.26	148.20	21.27	1.26	52.89	6.85	1.18	27.44	9.32
1.58	150.70	21.04	1.58	53.99	7.24	1.39	28.39	9.67
2.00	153.20	21.43	2.00	55.08	7.67	1.64	29.34	9.96
2.52	156.20	21.41	2.52	56.21	8.14	1.93	30.26	10.37
3.15	159.00	20.46	3.15	57.03	8.62	2.28	31.38	10.65
3.99	161.40	20.73	3.99	57.57	9.40	2.68	32.76	11.19
5.00	164.60	20.68	5.00	57.20	9.83	3.15	33.55	11.61
6.29	166.90	20.71	6.29	57.54	11.00	3.72	34.71	12.12
7.93	167.40	21.74	7.93	57.14	11.54	4.39	35.55	12.40
10.01	168.90	20.73	10.01	57.54	12.74	5.17	36.52	12.75
						6.10	37.58	12.99
						7.18	38.51	13.30
						8.48	39.57	13.91
						10.01	41.31	14.38

Table 12

xan-km she	ared gel (	0.5%	xan-km she	ared gel (	0.5%
conc.) in wa	ter		conc.) in 0.1	M ZnCl <sub>2</sub>	
frequency	G'	G''	frequency	G'	<b>G</b> ''
Hz	Pa	Pa	Hz	Pa	Pa
0.10	127.20	20.90	1.00	35.67	12.87
0.13	129.40	20.66	1.18	37.46	13.17
0.16	130.30	22.11	1.39	39.21	13.52
0.20	132.50	21.05	1.64	40.75	13.78
0.25	133.60	21.24	1.93	42.34	14.17
0.32	135.10	21.27	2.28	44.10	14.73
0.40	137.10	21.88	2.68	46.19	15.11
0.50	138.50	21.90	3.15	47.86	15.55
0.63	141.10	21.83	3.72	49.10	16.08
0.79	143.40	21.36	4.39	50.69	16.47
1.00	145.80	21.58	5.17	51.80	16.73

1.26	148.20	21.27	6.10	52.82	17.04
1.58	150.70	21.04	7.18	53.88	17.01
2.00	153.20	21.43	8.48	54.98	17.46
2.52	156.20	21.41	10.01	55.93	17.91
3.15	159.00	20.46			
3.99	161.40	20.73			
5.00	164.60	20.68			
6.29	166.90	20.71			
7.93	167.40	21.74			
10.01	168.90	20.73			

20% methanol at 70°C was added to a xanthan-km mixture at 70°C with stirring and was cooled (with and without stirring) to room temperature. The variation of G' and G" with frequency was compared for sheared and non-sheared gels in 20% methanol. The results are shown in Table 13. G' is greater than G" over the full frequency range for both the sheared and non-sheared gels and both are independent of frequency. G' values are higher for the xanthan-km non-sheared gels in 20% methanol than for the sheared gel. G' values obtained for the sheared gel containing 20% methanol are slightly greater than those obtained for the xanthan-km sheared gel in water. These results are shown in Table 14.

The variation of G' and G" as a function of temperature for a xanthan-km sheared gel on heating and cooling was also studied. The results are shown in Table 15. G' values become higher on cooling from 65 - 35°C. The sheared gel melts at ~ 62°C on heating and the onset of gelation on cooling also occurs at ~62°C. These values are similar to the values of melting and gelation obtained in water.

Table 13

xan-km sheared gel (0.5%			xan-km non-sheared gel				
conc.) in 20	% metha	nol	(0.5% conc.) in 20% methano				
frequency	G'	<b>G</b> ''	frequency	G'	G''		
Hz	Pa	Pa	Hz	Pa	Pa		
0.10	158.40	2.54	0.10	183.50	10.70		

	0.13	159.90	5.93	0.13	188.60	6.54
	0.16	160.50	3.25	0.16	189.00	8.49
	0.20	160.40	3.28	0.20	190.70	6.76
	0.25	162.30	6.02	0.25	190.30	7.52
5	0.32	162.60	5.70	0.32	193.60	6.84
	0.40	163.30	3.57	0.40	194.80	2.80
	0.50	164.20	4.37	0.50	194.50	4.26
	0.63	163.70	5.66	0.63	196.60	4.01
	0.79	164.20	4.42	0.79	194.40	3.22
10	1.00	167.10	4.81	1.00	196.90	5.99
	1.26	166.30	3.49	1.26	196.20	5.25
	1.58	167.50	5.97	1.58	195.80	3.54
	2.00	169.80	6.20	2.00	197.30	4.36
	2.52	168.00	7.13	2.52	197.80	5.14
15	3.15	170.50	7.01	3.15	197.40	3.44
	3.99	172.00	7.81	3.99	197.60	5.33
	5.00	171.80	8.78	5.00	198.90	5.35
	6.29	173.30	8.42	6.29	196.90	7.27
	7.93	172.30	9.58	7.93	190.00	13.16
20	10.01	170.60	10.16	10.01	184.80	15.58

Table 14

	xan-km she	xan-km sheared gel (0.5%			red gel (	0.5%
	conc.) in wa	ter		conc.) in 20%	% metha	nol
25	frequency	G'	G''	frequency	G'	G''
	Hz	Pa	Pa	Hz	Pa	Pa
	0.10	127.20	20.90	0.10	158.40	2.54
	0.13	129.40	20.66	0.13	159.90	5.93
	0.16	130.30	22.11	0.16	160.50	3.25
30	0.20	132.50	21.05	0.20	160.40	3.28
	0.25	133.60	21.24	0.25	162.30	6.02
	0.32	135.10	21.27	0.32	162.60	5.70
	0.40	137.10	21.88	0.40	163.30	3.57
	0.50	138.50	21.90	0.50	164.20	4.37
35	0.63	141.10	21.83	0.63	163.70	5.66
	0.79	143.40	21.36	0.79	164.20	4.42
	1.00	145.80	21.58	1.00	167.10	4.81
	1.26	148.20	21.27	1.26	166.30	3.49

1.58	150.70	21.04	1.58	167.50	5.97
2.00	153.20	21.43	2.00	169.80	6.20
2.52	156.20	21.41	2.52	168.00	7.13
3.15	159.00	20.46	3.15	170.50	7.01
3.99	161.40	20.73	3.99	172.00	7.81
5.00	164.60	20.68	5.00	171.80	8.78
6.29	166.90	20.71	6.29	173.30	8.42
7.93	167.40	21.74	7.93	172.30	9.58
10.01	168.90	20.73	10.01	170.60	10.16

Table 15

temp.	G'	G''	temp.	G'	G''	temp.	G'	G''
Deg C	Pa	Pa	Deg C	Pa	Pa	Deg C	Pa	Pa
35.00	152.30	6.79	47.70	104.40	11.09	52.70	82.63	7.49
35.30	146.40	8.22	48.00	105.80	6.90	52.90	80.60	6.86
35.70	148.10	7.82	48.30	103.60	6.60	53.30	77.23	6.01
36.00	146.40	8.26	48.60	102.20	8.62	53.60	74.57	6.13
36.30	146.60	8.10	48.90	99.69	10.82	61.70	10.05	4.87
36.60	144.80	9.38	49.20	100.90	7.99	62.00	8.55	4.72
36.80	139.70	12.88	49.60	99.55	8.35	62.40	7.36	4.46
37.10	140.30	8.39	49.90	97.87	9.18	62.60	6.24	4.20
37.40	139.80	8.38	50.20	97.44	7.45	63.00	5.22	3.94
37.80	138.80	8.85	50.50	94.80	9.24	63.30	4.38	3.72
38.20	136.20	6.66	50.80	93,17	6.38	63.60	3.78	3.59
38.40	136.60	7.12	51.10	91.73	7.06	64.00	3.35	3.48
38.80	135.40	8.41	51.40	90.05	7.10	64.20	3.15	3.41
39.00	133.70	8.26	51.70	88.96	7.80	64.60	3.12	3.40

1					07.05	(71	(4.00	3.06	2 34
1	47.10	108.00	8.93	52.00	87.85	6.74	64.90	3.00	3.34
	47.40	108.00	7.45	52.30	85.13	6.84	65.20	2.97	3.34

xan-km	(0.5% cond	c.) in	20%	methan	ol coolir	ıg cyc	le	· · · · · ·	
temp.	G'	G''					temp.	G'	<u>G''</u>
Deg C	Pa	Pa		Deg C	Pa	Pa	Deg C	Pa	Pa
65.20	3.44		3.86	54.70	44.01	2.99	44.60	136.70	5.3
64.80	3.49		3.86	54.40	45.90	3.08	44.40	139.80	4.10
64.50	3.48		3.90	54.10	48.99	2.94	44.00	143.10	5.72
64.20	3.50		3.91	53.80	52.35	2.48	43.70	147.30	5.4
63.90	3.54		3.92	53.50	55.36	2.04	43.40	151.60	4.7
63.60	3.63		3.93	53.10	57.11	2.78	43.10	151.00	5.2
63.30	3.66		3.98	52.80	60.35	2.36	42.70	154.40	4.9
62.90	3.76		4.03	52.50	63.38	2.56	42.40	154.10	3.3
62.70	3.85		3.96	52.20	64.06	2.60	42.10	156.70	7.7
62.30	3.97		4.01	51.90	67.19	2.27	41.80	159.20	
62.00	4.20		4.07	51.60	71.64	2.44	41.50	160.60	6.2
61.40	4.94		4.16	51.20	73.43	3.12	41.10	164.10	6.2
61.10	5.64		4.28	50.90	77.70	3.37	40.80	167.80	6.0
60.80	6.38	3	4.29	50.60	79.84	2.38	40.50	169.30	6.9
60.40	7.36	5	4.31	50.30	82.58	3.33	40.20	166.10	5.7
60.10	8.36	5	4.27	50.00	86.04	2.74	39.90	173.80	6.4
59.80	9.53	3	4.34	49.70	87.92	3.61	39.60	174.00	6.0
59.50	11.02	2	4.30	49.30	91.23	2.74	39.30	175.10	12.6
59.20	12.69	9	4.09	49.00	94.40	3.72	2 38.90	180.80	10.2
58.90	14.45	5	4.04	48.80	97.57	3.38	38.70	185.40	7.2
58.50	16.22	3	4.02	48.40	101.30	3.50	38.40	187.70	6.
58.20	17.92	2	3.87	7 48.10	104.90	6.20	38.00	189.50	7.4
57.90	20.00	0	3.93	47.80	110.60	5.09	37.70	184.00	5.
57.60	22.0	0	3.64	4 47.50	111.30	4.3	37.40	192.10	7.
57.30	24.5	2	3.53	3 47.20	116.4	0 3.7	37.10	195.50	6.5
57.00	26.4	7	3.39	9 46.80	119.5	0 3.6	36.80	197.70	7.2
56.70	28.9	3	3.0	3 46.50	123.2	0 4.2	8 36.50	197.90	7.9
56.30	31.1	1	3.3	7 46.20	126.5	0 5.4	6 36.30	199.40	6.
56.00	33.5	6	3.0	2 45.90	128.9	0 3.7	8 35.90	198.80	7.9
55.70	36.0	9	2.7	5 45.60	134.1	0 5.0	0 35.60	200.80	7.
55.40	38.7	7	2.9	6 45.30	133.7	0 5.9	2 35.30		
55.10	41.5	4	2.6	1 44.90	132.4	0 4.0	4 35.00	201.9	0 5.

Xanthan-km mixtures (0.5% 50:50) were prepared and adjusted to pH 2 and pH 10 while at ~70°C. The variation of G' and G" as a function of frequency for xanthan-km sheared gels at pH 5.5, at pH 2 and pH 10 was compared. The results are shown in Table 16. For xanthan-km sheared gels at pH 5.5 and at pH 10, G' is higher than G" at all frequency levels and neither are dependent on frequency. The G' values for the sheared gels at pH 5.5 and at pH 10 are very similar. However, for the xanthan-km stirred gel adjusted to pH 2, both moduli are frequency dependent and G" is slightly greater than G' over the frequency range 0.1 - 0.3 Hz where they cross over and G' becomes greater than G". This behaviour is typical of an entangled network and no gel-like characteristics are apparent.

Table 16

xan-km sh	eared ge	el	xan-km sh	eared g	el	xan-km	sheared	l gel
(0.5% cond	:.) @ pH	5.5	(0.5% con	c.) @ pl	H 2	(0.5% conc.) @ pH 10		
frequency G' G''		<b>G</b> ''	frequency G'		G''	frequency	G'	G''
Hz	Pa	Pa	Hz	Pa	Pa	Hz	Pa	Pa
0.10	127.20	20.90	0.10	2.16	2.51	0.10	123.60	4.73
0.13	129.40	20.66	0.13	2.49	2.81	0.13	122.60	4.24
0.16	130.30	22.11	0.16	2.87	3.12	0.16	125.10	4.64
0.20	132.50	21.05	0.20	3.25	3.44	0.20	125.00	4.32
0.25	133.60	21.24	0.25	3.79	3.82	0.25	125.80	5.07
0.32	135.10	21.27	0.32	4.32	4.21	0.32	127.20	2.35
0.40	137.10	21.88	0.40	4.94	4.57	0.40	126.90	5.35
0.50	138.50	21.90	0.50	5.53	5.00	0.50	127.50	5.04
0.63	141.10	21.83	0.63	6.23	5.44	0.63	128.50	5.23
0.79	143.40	21.36	0.79	6.99	5.87	0.79	130.00	3.05
1.00	145.80	21.58	1.00	7.87	6.30	1.00	129.20	5.86
1.26	148.20	21.27	1.26	8.77	6.81	1.26	131.20	5.44
1.58	150.70	21.04	1.58	9.65	7.34	1.58	131.20	
2.00	153.20	21.43	2.00	10.59	7.76	2.00	132.50	5.01
2.52	156.20	21.41	2.52	11.54	8.30	2.52	133.20	6.69
3.15	159.00	20.46	3.15	12.41	8.72	3.15	135.00	6.69

	3.99	161.40	20.73	3.99	13.13	9.32	3.99	136.40	6.86
ľ	5.00	164.60	20.68	5.00	13.51	9.79	5.00	137.90	8.20
ľ	6.29	166.90	20.71				6.29	139.30	8.32
ľ	7.93	167.40	21.74				7.93	136.90	9.60
t	10.01	168.90	20.73				10.01	141.10	9.42

Xanthan-km mixtures, (0.5% 50:50) were prepared by heating the polysaccharides to 90°C, 70°C and 50°C in water and electrolyte using the procedure outlined in Example 7. The variation of G' and G" for xanthan-km sheared gels at 25°C in water for the three mixing temperatures was studied as a function of frequency. The results are shown in Table 17. For all three samples G' is greater than G" over the whole frequency range. Samples heated to 90°C and 70°C were independent of frequency and had almost identical G' and G" values. However, a slight frequency dependence of the storage modulus over the entire frequency range was observed for the xanthan-km mixtures heated to 50°C indicating perhaps that the lower the temperature the xanthan-km mixtures are heated to, the lower the value of G' and the more frequency dependent it becomes therefore resulting in a decrease in gel strength.

The variations of G' and G" as a function of frequency for xanthan-km sheared gels at 25°C which were heated to 90°C, 70°C and 50°C in 0.15M NaCl were measured. The results are shown in Table 18. The gels show a typical gel-like response at all frequency levels studied. G' is always higher than G" but all three samples are slightly frequency dependent. The magnitude of G' is significantly less in 0.15M NaCl compared to water for samples prepared at 90°C and 70°C. At 50°C there is no noticeable reduction in G'.

The effect of mixing temperature on xanthan-km gels in 0.1M ZnCl<sub>2</sub> was also studied. G' and G" were measured as a function of frequency for xanthan-km sheared gels at 25°C which were heated to 90°C, 70°C and 50°C in 0.1M ZnCl<sub>2</sub>. The results are shown in Table 19. For all three samples G' is greater than G" over the frequency range studied but all three show a slight frequency dependence. G' and G" values are only slightly higher for the sample heated to 90°C in comparison to those

heated to 70°C and 50°C.

Table 17

xan-km sho	eared gel	(0.5%	xan-km sh	eared ge	el	xan-km sh	eared	gel	
conc.) in w	ater heat	ed to	(0.5% con	c.) in wa	ter	(0.5% conc.) in water			
90° <b>C</b>			heated to 70°C			heated to 50°C			
frequency	G'	G''	frequency	G'	G''	frequency	G'	G''	
Hz	Pa	Pa	Hz	Pa	Pa	Hz	Pa	Pa	
0.10	127.20	20.90	0.10	133.60	14.52	0.10	39.88	11.94	
0.13	129.40	20.66	0.13	135.70	15.19	0.13	42.16	11.9	
0.16	130.30	22.11	0.16	136.60	15.50	0.16	43.93	11.90	
0.20	132.50	21.05	0.20	137.80	15.42	0.20	45.53	11.84	
0.25	133.60	21.24	0.25	138.00	16.12	0.25	47.49	11.9	
0.32	135.10	21.27	0.32	139.70	16.12	0.32	48.60	12.2	
0.40	137.10	21.88	0.40	140.00	16.34	0.40	50.09	11.9	
0.50	138.50	21.90	0.50	141.40	16.15	0.50	52.01	11.9	
0.63	141.10	21.83	0.63	142.70	16.51	0.63	53.47	12.0	
0.79	143.40	21.36	0.79	144.00	16.87	0.79	54.67	12.4	
1.00	145.80	21.58	1.00	145.50	17.15	1.00	56.24	12.5	
1.26	148.20	21.27	1.26	147.40	17.78	1.26	58.08	12.7	
1.58	150.70	21.04	1.58	149.50	18.18	1.58	59.94	13.1	
2.00	153.20	21.43	2.00	151.70	18.73	2.00	61.73	13.1	
2.52	156.20	21.41	2.52	154.20	19.41	2.52	63.83	13.7	
3.15	159.00	20,46	3.15	156.60	19.41	3.15	65.39	13.9	
3.99	161.40	20.73	3.99	159.10	20.36	3.99	67.16	14.4	
5.00	164.60	20.68	5.00	161.90	21.28	5.00	68.50	15.1	
6.29	166.90	20.71	6.29	164.20	21.69	6.29	69.99	15.2	
7.93	167.40	21.74	7.93	165.40	24.51	7.93	71.43	15.7	
10.01	168.90	20.73	10.01	166.70	22.71	10.01	74.41	16.2	

Table 18

xan-km sh	eared	gel in	xan-km sh	eared	gel in	xan-km sh	eared	gel (0.5%	
0.15M NaCl heated to			0.15M NaC	CI hea	ted to	conc.) in 0.15M NaCl			
90°C			70°C			heated to 50°C			
frequency	G'	G''	frequency	frequency G' G''		frequency	G'	<b>G</b> ''	
Hz	Hz Pa Pa		Hz	Pa	Pa	Hz	Pa		

					_				
ſ	0.10	25.87	4.31	0.10	19.81	4.54	0.10	28.28	2.89
Ī	0.13	26.65	4.42	0.13	21.42	4.34	0.13	28.79	2.96
Ī	0.16	27.74	4.44	0.16	22.19	4.31	0.16	29.45	3.06
	0.20	28.41	4.96	0.20	23.12	4.36	0.20	30.22	3.32
5	0.25	29.20	5.21	0.25	23.74	4.86	0.25	30.41	3.31
	0.32	30.16	5.22	0.32	24.38	5.29	0.32	30.85	3.24
	0.40	30.82	5.87	0.40	25.19	5.86	0.40	31.42	3.63
	0.50	31.78	6.02	0.50	26.00	5.86	0.50	31.98	4.06
	0.63	32.65	6.34	0.63	26.89	6.04	0.63	32.72	4.17
10	0.79	33.49	6.92	0.79	27.73	6.46	0.79	33.09	4.40
	1.00	34.48	7.14	1.00	28.67	6.93	1.00	33.78	4.70
	1.26	35.49	7.69	1.26	29.53	7.43	1.26	34.15	5.09
	1.58	36.71	8.19	1.58	30.41	8.15	1.58	34.86	5.45
	2.00	37.92	8.75	2.00	31.43	8,62	2.00	35.54	5.96
15	2.52	38.79	9.19	2.52	32.59	8.90	2.52	35.66	6.16
	3.15	39.88	9.78	3.15	33.75	9.56	3.15	36.17	6.93
	3.99	40.70	10.22	3.99	34.75	9.86	3.99	35.83	7.76
	5.00	41.90	10.99	5.00	35.52	10.79	5.00	36.13	8.25
	6.29	42.61	11.71	6.29	36.55	11.24	6.29	35.64	8.76
20	7.93	44.20	12.49	7.93	38.09	12.02	7.93	35.32	9.42
	10.01	46.49	13.19	10.01	40.04	12.71	10.01	35.14	10.40
			-						

# Table 19

ļ	xan-km sh	eared g	gel in	xan-km sh	eared	gel in	xan-km sheared gel in 0.1M ZnCl <sub>2</sub> heated to			
25	0.1M ZnC	l <sub>2</sub> heate	d to	0.1M ZnC	l <sub>2</sub> heat	ed to				
	90°C			70°C			50°C			
	frequency	G'	G''	frequency	G'	G''	frequency	G'	G''	
	Hz	Pa	Pa	Hz	Pa	Pa	Hz	Pa	Pa	
,	1.00	35.67	12.87	1.00	33.38	8.00	1.00	32.75	7.57	
30	1.18	37.46	13.17	1.26	34.65	8.28	1.26	33.68	7.92	
	1.39	39.21	13.52	1.58	35.47	8.59	1.58	34.91	8.18	
	1.64	40.75	13.78	2.00	36.47	9.04	2.00	35.87	8.57	
	1.93	42.34	14.17	2.52	37.80	9.56	2.52	36.95	8.75	
	2.28	44.10	14.73	3.15	38.90	10.10	3.15	38.01	8.97	
35	2.68	46.19	15.11	3.99	39.65	10.08	3.99	39.03	9.63	
	3.15	47.86	15.55	5.00	40.00	11.02	5.00	39.29	9.83	
	3.72	49.10	16.08	6.29	40.77	11.17	6.29	40.28	9.99	

- 1									
1	4.39	50.69	16.47	7.93	41.38	11.68	7.93	40.98	10.21
	5.17	51.80	16.73	10.01	42.26	12.40	10.01	42.62	10.63
	6.10	52.82	17.04						
	7.18	53.88	17.01						
	8.48	54.98	17.46						
	10.01	55.93	17.91						

The following examples illustrate formulations in which gels of the present invention can be used:

The gel used in these formulation examples was provided in the form of a powder containing xanthan gum, locust bean gum, konjac mannan gum and sodium chloride.

The powder was incorporated in the aqueous phase of the formulation. The aqueous phase was heated to at least 65°C, thoroughly mixed and then cooled with constant stirring to below 35°C. A gel of the invention was thereby formed in situ in the composition.

# Formulation Example 1 - Bath Gel (1)

		Sample 1	Sample 2
20		%w/w	%w/w
	Sodium laureth sulfate	46.5	46.3
	Disodium pareth-3 sulfosuccinate	25	25
	Lauric fatty acids	11.5	11.5
	Diethanolamine	11.5	11.5
25	Methyl gluceth-10 ethoxylated	2.5	2.5
	Laneth-16	3	3
	Gel of the invention	-	0.3
	Preservative	q.s	q.s
	Parfum	q.s	q.s

Method:

Stage 1

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The following materials were added to water in order using stirring:

Sodium laureth sulfate, Disodium pareth sulfosuccinate, lauric fatty acids,
diethanolamine, methyl gluceth-10, laneth-16 (and gel of the invention in sample 2).

Stage 2

Maintaining the stirring, the bulk was heated to 65°C. when uniform, the bulk was cooled with constant stirring to below 35°C.

#### Stage 3

The preservative and perfume were added and the product was made to weight with purified water. The product was stirred until cool and uniform.

10

Advantages of using a gel of the invention:

- -Stable, reproducible viscosity build.
- -Improves the pouring, causing no drips.
- -Improves textural properties.
- 15 -Results in a conditioned end feel.

# Formulation Example 2 - Bath Gel (2)

		Sample 1	Sample 2
		%w/w	%w/w
20	Aqua	76	75.8
	Sodium lauryl sulfate	20	20
	Cocamidopropyl betaine	1.5	1.5
	Sodium chloride	0.2	0.2
	PEG-6 Cocamide	1	1
25	Dipropylene glycol	0.25	0.25
	PEG-18 glyceryl oleate/cocoate	0.5	0.5
	Gel of the invention	-	0.2
	Preservative	q.s	q.s
	Parfum	q.s	q.s
30		Ì	

Method:

# Stage 1

The following materials were added to the water in order using stirring:

Sodium lauryl sulfate, sodium chloride, PEG-6 cocamide, dipropylene glycol, PEG-

18, cocamidopropyl betaine (and gel of the invention in sample 2).

# 5 Stage 2

Maintaining stirring, the bulk was heated to 65°C. Once uniform, the bulk was cooled with constant stirring to below 35°C.

# Stage 3

The preservative and perfume were added and the product was made to weight with purified water. The product was stirred until cool and uniform.

Advantages of using a gel of the invention:

- -Stable, reproducible viscosity build.
- -Improves the pouring, causing no drips.
- 15 -Improves textural properties.
  - -Results in a conditioned end feel.

# Formulation Example 3 - Shower Gel (1)

		Sample 1	Sample 2
20		%w/w	%w/w
	Aqua	77.5	77.3
	Sodium lauryl sulfate	10.5	10.5
	Cocoamphocarboxypropionate	6	6
	Sodium lauroyl sarcosinate	3	3
25	Cocamide MEA	3	3
	Gel of the invention	-	0.2
	Preservative	q.s	q.s
	Parfum		q.s

#### 30 Method:

Stage 1

The following materials were added to the water in order with stirring:

Sodium lauryl sulfate, cocoamphocarboxypropionate, sodium lauroyl sarcosinate,
cocamide MEA (and gel of the invention in sample 2).

#### Stage 2

Maintaining stirring, the bulk was heated to 65°C. Once uniform, the bulk was cooled with constant stirring to below 35°C.

#### Stage 3

The preservative and perfume were added and the product was made to weight with purified water. The product was stirred until cool and uniform.

10

Advantages of using a gel of the invention:

- -Stable, reproducible viscosity build.
- -Improves the pouring, causing no drips.
- -Rheology modifier.
- 15 -Improves textural properties.
  - -Results in a conditioned end feel.

# Formulation Example 4 - Shower Gel (2)

		Sample 1	Sample 2
20		%w/w	%w/w
	Aqua	75.5	75.3
	Sodium lauryl sulfate	20	20
	Cocamidopropyl betaine	1.5	1.5
	Sodium chloride	0.2	0.2
25	PEG-6 Cocamide	1	1
	Dipropylene glycol	0.25	0.25
	PEG-18 glyceryl oleate/cocoate	0.5	0.5
	PEG-40 hydrogenated castor oil	0.5	0.5
	Polyquaternium-7	0.04	-
30	PEG-7 glyceryl cocoate	0.5	0.5
	Tetrasodium EDTA	0.02	0.02

Gel of the invention	-	0.2
Preservative	q.s	q.s
Parfum	q.s	q.s

#### 5 Method:

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#### Stage 1

The following materials were added to the water in order with stirring: (in sample 1, polyquaternium-7 was added first) Tetrasodium EDTA, Sodium lauryl sulfate, sodium chloride, PEG-6, dipropylene glycol, PEG-18, PEG-40, PEG-7, cocamidopropyl betaine (and gel of the invention in sample 2).

#### Stage 2

Maintaining stirring, the bulk was heated to 65°C. Once uniform, the bulk was cooled with constant stirring to below 35°C.

Stage 3

The preservative and perfume were added and the product was made to weight with purified water. The product was stirred until cool and uniform.

Advantages of using a gel of the invention:

- -Stable, reproducible viscosity build.
- 20 -Improves the pouring, causing no drips.
  - -Rheology modifier.
  - -Improves textural properties.
  - -Skin conditioning effect.
  - -Results in a conditioned end feel.

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# Formulation Example 5 - Shampoo (1)

		Sample 1	Sample 2
		%w/w	%w/w
	Aqua	84.86	84.64
30	Ammonium lauryl sulfate	5.04	5.04
	Lauroyl sarcosine	5	5

	Lauramide DEA	5	5
	Tetrasodium EDTA	0.1	0.1
	Gel of the invention	-	0.2
	Preservative	q.s	q.s
5	Parfum	q.s	q.s

#### Stage 1

The following materials were added to the water in order with stirring:

Tetrasodium EDTA, Ammonium lauryl sulfate, lauroyl sarcosinate, lauramide DEA (and gel of the invention in sample 2).

#### Stage 2

Maintaining stirring, the bulk was heated to 65°C. Once uniform, the bulk was cooled with constant stirring to below 35°C.

# 15 Stage 3

The preservative and perfume were added and the product was made to weight with purified water. The product was stirred until cool and uniform.

Advantages of using a gel of the invention:

- 20 -Stable, reproducible viscosity build.
  - -Improves the pouring, causing no drips.
  - -Rheology modifier.
  - -Soft hair-feel effect.
  - -Improves textural properties.
- -Results in a conditioned end feel.

# Formulation Example 6 - Shampoo (2)

		Sample 1	Sample 2
		%w/w	%w/w
30	Aqua	87.5	87.3
	Sodium lauryl sulfate	7.8	7.8

	Sodium chloride	0.8	0.8
	Cocamidopropyl betaine	0.75	0.75
	Glycol disterate	0.8	0.8
	Laureth-3	2	2
5	Gel of the invention	-	0.2
	Preservative	q.s	q.s
	Parfum	q.s	q.s

# 10 Stage 1

The following materials were added to the water in order with stirring: Sodium lauryl sulfate, sodium chloride, laureth-3, cocamidopropyl betaine (and gel of the invention in sample 2).

### Stage 2

Maintaining stirring, the bulk was heated to 65°C. Glycol distearate was then added and dispersed. Once uniform, the bulk was cooled with constant stirring to below 35°C.

#### Stage 3

The preservative and perfume were added and the product was made to weight with purified water. The product was stirred until cool and uniform and the pearl had set up.

Advantages of using a gel of the invention:

- -Stable, reproducible viscosity build.
- 25 -Improves the pouring, causing no drips.
  - -Rheology modifier.
  - -Soft hair-feel effect.
  - -Pearl stabiliser.
  - -Improves textural properties.
- -Results in a conditioned end feel.

# Formulation Example 7 - Shampoo and Conditioner 2-in-1 (1)

		Sample 1	Sample 2
		%w/w	%w/w
	Aqua	67.9	67.6
5	Alpha olefin sulfonate	25	25
	Cocamide DEA	2	2
	Lauramide DEA	1	1
	Oleamide MIPA	1.5	1.5
	Cocamidopropyl betaine	2	2
10	Oleth-3 phosphate	0.1	0.1
	Lauric acid	0.25	0.25
	Sodium chloride	0.25	0.25
	Gel of the invention	-	0.3
	Preservative	q.s	q.s
15	Parfum	q.s	q.s

#### Method:

# Stage 1

The following materials were added to the water in order with stirring:

Alpha olefin sulfonate, cocamide DEA, lauramide DEA, oleamide MIPA, cocamidopropyl betaine, lauric acid, oleth-3 phosphate (and Gel of the invention in sample 2).

#### Stage 2

Maintaining stirring, the bulk was heated to 65°C. Once uniform, the bulk was cooled with constant stirring to below 35°C.

#### Stage 3

25

The preservative and perfume were added and the product was made to weight with purified water. The product was stirred until cool and uniform.

- Advantages of using a gel of the invention:
  - -Stable, reproducible viscosity build.

- -Improves the pouring, causing no drips.
- -Rheology modifier.
- -Soft hair-feel effect.
- -Stabiliser.
- 5 -Improves textural properties.
  - -Results in a conditioned end feel.

# Formulation Example 8 - Shampoo and Conditioner 2-in-1 (2)

		Sample 1	Sample 2
10		%w/w	%w/w
	Aqua	83	83.1
	Sodium lauryl sulfate	8	8
	Cocamidopropyl betaine	2	2
	Ethylene glycol monostearate	3	3
15	Dimethicone	3.5	3.5
	Hydroxypropyl guar hydroxypropyltrimonium chloride	0.4	-
	Citric acid	0.02	0.02
	Tetrasodium EDTA	0.05	0.05
	Gel of the invention	-	0.3
20	Preservative	q.s	q.s
	Parfum	q.s	q.s

#### Method:

# Stage 1

The following materials were added to the water in order with stirring:

(hydroxypropyl guar in was added first in sample 1) Citric acid, Tetrasodium EDTA, sodium lauryl sulfate, cocamidopropyl betaine, ethylene glycol monostearate (and gel of the invention in sample 2).

#### Stage 2

Maintaining stirring, the bulk was heated to 75°C. Once uniform, dimethicone was added and dispersed. The bulk was then cooled with constant stirring to below 35°C.

# Stage 3

The preservative and perfume were added and the product was made to weight with purified water. The product was stirred until cool and uniform.

- 5 Advantages of using a gel of the invention:
  - -Stable, reproducible viscosity build.
  - -Improves the pouring, causing no drips.
  - -Rheology modifier.
  - -Soft hair-feel effect.
- 10 -Stabilises dimethicone emulsion.
  - -Improves textural properties.
  - -Results in a conditioned end feel.

# Formulation Example 9 - Anti-dandruff Shampoo (1)

15		Sample 1	Sample 2
		%w/w	%w/w
	Aqua	77.35	76.95
	Sodium N-methyl-N-myristoyl-taurate	13.65	13.65
	Laneth-10 acetate	1	1
20	Coconut oil diethanolamine condensate	5	5
	Zinc pyrithione (dispersion)	2	2
	Gel of the invention	-	0.4
	Preservative	q.s	q.s
	Parfum	q.s	q.s

25

Method:

Stage 1

The following materials were added to the water in order with stirring:

Sodium methyl myristoyl taurate, laneth-10, coconut oil, zinc pyrithione dispersion

30 (and gel of the invention in sample 2).

Stage 2

Maintaining stirring, the bulk was heated to 65°C. Once uniform, the bulk was cooled with constant stirring to below 35°C.

#### Stage 3

5

The preservative and perfume were added and the product was made to weight with purified water. The product was stirred until cool and uniform.

Advantages of using a gel of the invention:

- -Stable, reproducible viscosity build.
- -Improves the pouring, causing no drips.
- 10 -Rheology modifier.
  - -Soft hair-feel effect.
  - -Suspension of Zinc Pyrithione.
  - -Aids deposition.
  - -Improves textural properties.
- -Results in a conditioned end feel.

# Formulation Example 10 - Anti-dandruff Shampoo (2)

		Sample 1	Sample 2
		%w/w	%w/w
20	Aqua	83	83
	Sodium lauryl sulfate	8	8
	Cocamidopropyl betaine	2	2
	Ethylene glycol monostearate	3	3
	Dimethicone	3.5	3.5
25	Hydroxypropyl guar hydroxypropyltrimonium chloride	0.4	-
	Citric acid	0.02	0.02
	Tetrasodium EDTA	0.05	0.05
	Zinc pyrithione (dispersion)	1	1
	Gel of the invention	-	0.4
30	Preservative	q.s	q.s
	Parfum	q.s	q.s

#### Stage 1

The following materials were added to the water in order with stirring:

(hydroxypropyl guar in was added first in sample 1) Citric acid, Tetrasodium EDTA, sodium lauryl sulfate, cocamidopropyl betaine, ethylene glycol monostearate, zinc pyrithione dispersion (and gel of the invention in sample 2).

#### Stage 2

Maintaining stirring, the bulk was heated to 75°C. Once uniform, dimethicone was added and dispersed. The bulk was then cooled with constant stirring to below 35°C. Stage 3

The preservative and perfume were added and the product was made to weight with purified water. The product was stirred until cool and uniform.

- 15 Advantages of using a gel of the invention:
  - -Stable, reproducible viscosity build.
  - -Improves the pouring, causing no drips.
  - -Rheology modifier.
  - -Soft hair-feel effect.
- 20 -Stabilises dimethicone emulsion.
  - -Suspends zinc pyrithione.
  - -Improves textural properties.
  - -Results in a conditioned end feel.

# 25 <u>Formulation Example 11</u> - Liquid Soap

		Sample 1	Sample 2
		%w/w	%w/w
	Aqua	83.7	84.4
	Magnesium aluminium silicate	1	-
30	Potassium hydroxide	2	2
	Propylene glycol	2.5	2.5

	Sodium laureth sulfate	1.8	1.8
	Oleic acid	9	9
	Gel of the invention	-	0.3
	Preservative	q.s	q.s
5	Parfum	q.s	q.s

#### Stage 1

In sample 1, magnesium aluminium silicate was added to the water and dispersed using stirring. In both samples, potassium hydroxide was added and dispersed.

Propylene glycol, sodium laureth sulfate and oleic acid were then added (and gel of the invention in sample 2).

#### Stage 2

Maintaining the stirring, the bulk was heated to 75°C, giving enough time for the oleic acid to saponify. The bulk was then cooled with constant stirring to below 35°C.

#### Stage 3

The preservative and perfume were added and the product was made to weight with purified water. The product was stirred until cool and uniform.

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Advantages of using a gel of the invention:

- -Stable, reproducible viscosity build.
- -Rheology modifier.
- -Skin conditioning effect.
- 25 -Stabilises the pearl.
  - -Improves textural properties.
  - -Results in a conditioned end feel.

# Formulation Example 12 - Beaded Shower Gel

Sample 1 Sample 2 %w/w %w/w

	Aqua	77	82.6
	Sodium lauryl sulfate	9.5	9.5
	Citric acid	0.02	0.02
	Tetrasodium EDTA	0.05	0.05
5	Mica	0.1	0.1
	Acrylates copolymer	6	-
	PEG-40 hydrogenated castor oil	0.5	0.5
	PEG-6 Cocamide	1	1
	Dipropylene glycol	0.25	0.25
10	PEG-18 glyceryl oleate/cocoate	0.5	0.5
	Gel of the invention	-	0.4
	Preservative	q.s	q.s
	Parfum	q.s	q.s

20

# Stage 1

The following materials were added to the water in order with stirring:

Citric acid, Tetrasodium EDTA, sodium lauryl sulfate, PEG-40, PEG-6, PEG-18,

(acrylates copolymer in sample 1) and dipropylene glycol (and gel of the invention in sample 2).

#### Stage 2

Maintaining stirring, the bulk was heated to 70°C. Once uniform, the bulk was then cooled with constant stirring to below 35°C.

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#### Stage 3

The preservative, perfume and mica were added and the product was made to weight with purified water. The product was stirred until cool and uniform.

Advantages of using a gel of the invention:

- -Stable, reproducible viscosity build.
- 30 -Improves the pouring, causing no drips.
  - -Rheology modifier.

- -Skin conditioning effect.
- -Suspending beads.
- -Improves textural properties.
- -Results in a conditioned end feel.

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# Formulation Example 13 - Oil-in-water emulsion (1)

		Sample 1	Sample 2
		%w/w	%w/w
	Aqua	63	63
10	Carbomer 940	0.35	-
	1,3-Butylene glycol	2	2
	Tetrasodium EDTA	0.05	0.05
	Potassium Hydroxide	0.06	0.06
	Polysorbate 80	1	1
15	Paraffinum liquidum	18	18
	Gel of the invention	-	0.35
	Preservative	q.s	q.s

#### Method:

# 20 Stage 1

25

EDTA was dispersed in the water using stirring. For sample 1, the Carbomer was then added and hydrated using homogenisation for 30 minutes. For sample 2, the gel of the invention was added and dispersed using stirring.

Butylene glycol and Polysorbate 80 were then added and mixed until uniform using stirring. This phase was then heated to 70°C.

#### Stage 2

The paraffinum liquidum was weighed into a separate vessel and heated to 70°C. Stage 3

The oil phase was added to the aqueous phase and an emulsion was formed using high shear homogenisation for 10 minutes. The Potassium Hydroxide was then added and the shear was maintained for a further 5 minutes.

# Stage 4

The emulsion was cooled to below 35°C using stirring. The preservative was then added and the product was made to weight with purified water. The emulsion was stirred until cool and uniform.

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Advantages of using a gel of the invention:

- -Stable, reproducible viscosity build.
- -Rheology modifier.
- -Emulsion stabiliser.
- 10 -Improves textural properties.
  - -Skin conditioning effect.
  - -Results in a conditional end feel.

# Formulation Example 14 - Oil-in-water emulsion (2)

	•	,	
15		Sample 1	Sample 2
		%w/w	%w/w
	Aqua	77	77
	Glycerin	5	5
	Paraffinium liquidum	5	5
20	Dicaprylyl maleate	3	3
	Petrolatum	3	3
	Cetyl alcohol	2	2
	Steareth-2	1.5	1.5
	Glyceryl stearate	1.5	1.5
25	Steareth-21	1	1
	Sodium citrat	0.06	0.06
	Citric acid	0.02	0.02
	Hydroxyethyl cellulose	0.3	_
	Tetrasodium EDTA	0.05	0.05
30	Gel of the invention	! -	0.3
	Preservative	q.s	q.s

q.s

Parfum q.s

Method:

Stage 1

5 EDTA, citric acid, sodium citrate and glycerin were dispersed in the water using stirring. For sample 1, the hydroxyethyl cellulose was then added and hydrated using homogenisation for 5 minutes. For sample 2, the gel of the invention was added and dispersed with stirring. This phase was then heated to 70°C.

Stage 2

The oils and waxes were mixed in a separate vessel and heated to 70°C until melted.

Stage 3

The oil phase was added to the aqueous phase and an emulsion was formed using high shear homogenisation for 10 minutes.

Stage 4

The emulsion was cooled to below 35°C using stirring. The preservative was then added and the product was made to weight with purified water. The emulsion was stirred until cool and uniform.

Advantages of using a gel of the invention:

- -Stable, reproducible viscosity build.
  - -Rheology modifier.
  - -Skin conditioning effect.
  - -Emulsion stabiliser.
  - -Improves textural properties.
- 25 -Results in a conditioned end feel.

#### Formulation Example 15 - Water-in-oil emulsion

		Sample I	Sample 2
		%w/w	%w/w
30	Aqua	76.93	76.73
	1,3-Butylene glycol	5	5

	Sodium chloride	1	1
	Cetyl dimethicone copolyol	1.5	1.5
	Sorbitan isostearate	0.5	0.5
	Stearic acid	0.05	0.05
5	Paraffinum liquidum	15	15
	Butylated hydroxytoluene	0.02	0.02
	Gel of the invention	-	0.2
	Preservative	q.s	q.s

Stage 1

Sodium chloride, butylene glycol (and gel of the invention in sample 2) were added to the water using stirring. This phase was heated to 70°C maintaining stirring.

Stage 2

The oil phase was mixed and heated to 70°C until melted and uniform.

Stage 3

Using high speed stirring, the aqueous phase was slowly added to the oil phase and stirred until emulsified and uniform. The emulsion was then transferred to a homogeniser and high shear was applied for 5 minutes.

#### 20 Stage 4

The emulsion was cooled to below 35°C with stirring and the preservative was added. Stirring continued until cool and uniform.

Advantages of using a gel of the invention:

- 25 -Improves textural properties.
  - -Results in a conditioned end feel.

# Formulation Example 16 - Cold Cream

%w/w

69	68.7
1	-
1.5	1.5
1.5	1.5
20	20
3.5	3.5
3.5	3.5
-	0.3
q.s	q.s
	1 1.5 1.5 20 3.5 3.5

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#### Method:

#### Stage 1

In sample 1, magnesium aluminium silicate was added to the water and dispersed using stirring, followed by the addition and dispersion of polysorbate 60. In sample 2, the gel of the invention was added and dispersed using stirring. This phase was heated to 70°C maintaining stirring.

#### Stage 2

The oil phase was mixed in a separate vessel and heated to 70°C until melted and uniform.

# 20 Stage 3

Maintaining the temperature of both phases, the oil phase was added to the aqueous phase and an emulsion was formed using high shear homogenisation for 10 minutes. Stage 4

The emulsion was cooled to below 35°C using stirring. The preservative was then added and the product was made to weight with purified water. The emulsion was stirred until cool and uniform.

Advantages of using a gel of the invention:

- -Stable, reproducible viscosity build.
- 30 -Rheology modifier.
  - -Emulsion stabiliser.

- -Improves textural properties.
- -Skin conditioning effect resulting in a conditioned end feel.

# Formulation Example 17 - Anti-perspirant Stick

5		Sample 1	Sample 2
		%w/w	%w/w
	Aqua	32.5	32.2
10	Stearic acid	6	6
	Sorbitol	2.8	2.8
	Sodium aluminium chlorhydroxylactate	18	18
	Sodium hydroxide	0.7	0.7
	Ethanol	40	40
	Gel of the invention	-	0.3

#### 15 Method:

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#### Stage 1

Into the water, stearic acid, sorbitol (and gel of the invention in sample 2) were added. Sodium hydroxide was then added with stirring. This was heated to 70°C until uniform, and giving the stearic acid sufficient time to saponify. The sodium aluminium chlorhydroxylacetate was added and dispersed with stirring.

#### Stage 2

The bulk was cooled to 50°C with stirring. At this temperature, the ethanol was added and dispersed with stirring. The cooling continued until the bulk was at ambient temperature.

This product was packed into solvent sealed cases.

Advantages of using a gel of the invention:

- -Sodium stearate crystal structure modifier.
- -Rheology modifier.
- 30 -Moisture absorption properties.
  - -Improves textural properties.

#### -Results in a conditioned end feel.

# Formulation Example 18 - Body Lotion

		Sample 1	Sample 2
5		%w/w	%w/w
	Aqua	68.47	68.47
	Carbomer 940	0.3	-
	Sodium hydroxide	0.028	0.028
	Glycerine	5	5
10	Glyceryl monostearate and polyoxyethylene	4	4
	Stearate fatty acid ester	2	2
	Paraffinum liquidum	15	15
	Cholesterol	5	5
	Oleyl alcohol	2	2
15	Tetrasodium EDTA	0.05	0.05
	Gel of the invention	-	0.3
	Preservative	q.s	q.s

#### Method:

# 20 Stage 1

25

EDTA was dispersed in the water using stirring. For sample 1, the Carbomer was then added and hydrated using homogenisation for 30 minutes. For sample 2, the gel of the invention was added and dispersed using stirring.

Glycerin was then added and stirred until uniform using stirring. This phase was then heated to 70°C.

#### Stage 2

The oil phase was mixed in a separate vessel and heated to 70°C until melted and uniform.

#### Stage 3

The oil phase was added to the aqueous phase and an emulsion was formed using high shear homogenisation for 10 minutes. Sodium hydroxide was then added and

the shear was maintained for a further 5 minutes.

# Stage 4

5

The emulsion was cooled to below 35°C using stirring. The preservative was then added and the product was made to weight with purified water. The emulsion was stirred until cool and uniform.

Advantages of using a gel of the invention:

- -Stable, reproducible viscosity build.
- -Rheology modifier.
- 10 -Emulsion stabiliser.
  - -Improves textural properties.
  - -Skin conditioning effect.
  - -Results in a conditioned end feel.

# 15 <u>Formulation Example 19</u> - Hair Conditioner (1)

		Sample 1	Sample 2
		%w/w	%w/w
	Aqua	89.8	89.5
	Cetyl trimethyl ammonium chloride	1.5	1.5
20	Alumina	0.5	0.5
	Petrolatum	1.5	1.5
	Glyceryl stearate	0.2	0.2
	Acetylated lanolin alcohol	2	2
	Mineral oil (and) lanolin alcohol	2	2
25	Stearyl alcohol	2.5	2.5
	Gel of the invention	-	0.3
	Preservative	q.s	q.s
	Parfum	q.s	q.s

!

30 Method:

Stage 1

The materials were added to the water in the order shown above (excluding the parfum and preservative) with stirring. Gel of the invention was added at this stage in sample 2. With constant stirring, the bulk was heated to 70°C until all materials were dispersed, melted and uniform.

# 5 Stage 2

The product was cooled to below 35°C using stirring. The preservative was then added and the product was made to weight with purified water. The product was then stirred until cool and uniform.

# Advantages of using a gel of the invention::

- -Stabiliser
- -Conditioning agent
- -Rheology modifier
- -Deposition aid
- 15 -Improves textural properties
  - -Results in a conditioned end feel.

# Formulation Example 20 - Hair Conditioner (2)

		Sample 1	Sample 2
20		%w/w	%w/w
	Aqua	90	90.1
	Cetyl alcohol	4	4
	Cocamide MEA	2	2
	Stearamidopropyl dimethylamine	1	1
25	Centrimonium chloride	0.5	0.5
	Citric acid	0.02	0.02
	Lactic acid	0.4	0.4
	Hydroxyethyl cellulose	0.5	_
	Tetrasodium EDTA	0.05	0.05
30	Gel of the invention	_	0.4
	Preservative	q.s	q.s

Parfum q.s q.s

Method:

# 5 Stage 1

EDTA, citric acid and lactic acid were added to the water and dispersed with stirring. In sample 1, the hydroxyethyl cellulose was added and hydrated using a homogeniser for 5 minutes. In sample 2, the gel of the invention was added with stirring.

The other materials were added to the water in the order shown above (excluding the parfum and preservative) with stirring. With constant stirring, the bulk was heated to 70°C until all materials were dispersed, melted and uniform.

#### Stage 2

15

The product was cooled to below 35°C using stirring. The preservative was then added and the product was made to weight with purified water. The product was then stirred until cool and uniform.

Advantages of using a gel of the invention::

- -Stabiliser
- -Conditioning agent
- 20 -Rheology modifier
  - -Deposition aid
  - -Improves textural properties
  - -Results in a conditioned end feel.

# 25 <u>Formulation Example 21</u> - Fluid Foundation (1)

		Sample 1	Sample 2	
		%w/w	%w/w	
	Aqua	61.5	61.6	
	Carbomer 941	0.5	-	
30	Lanolin oil	5	5	
	Paraffinum liquidum	3.5	3.5	

	Stearyl alcohol (and) ceteareth-20 condensate	3	3
	Triethanolamine	0.5	0.5
	Ethanol	26	26
	Tetrasodium EDTA	0.02	0.02
5	Gel of the invention		0.4
	Pigments	q.s	q.s
	Preservative	q.s	q.s

### 10 Stage 1

EDTA was added to the water and dispersed with stirring. In sample 1, the Carbomer was added and hydrated using a homogeniser for 30 minutes. In sample 2, the gel of the invention was added with stirring.

This phase was then heated to 70°C.

# 15 Stage 2

The oil phase was mixed in a separate vessel and heated to 70°C until melted and uniform.

#### Stage 3

The oil phase was added to the aqueous phase and an emulsion was formed using high shear homogenisation for 10 minutes. Sodium hydroxide was then added and the shear was maintained for a further 5 minutes. Maintaining the shear, the pigments were slowly added and dispersed. The emulsion was sheared until uniform.

#### Stage 4

The emulsion was then cooled to below 35°C with stirring. The preservative was added and the product was made to weight with purified water.

Advantages of using a gel of the invention:

- -Dispersing aid for the pigments
- -Suspending the pigments
- 30 -Emulsion stabiliser
  - -Rheology modifier

- -Improves textural properties
- -Results in a conditioned end feel.

# Formulation Example 22 - Fluid Foundation (2)

5		Sample 1	Sample 2
		%w/w	%w/w
	Aqua	72	72
	1,3-butylene glycol	8	8
	Glycerin	5	5
10	Xanthan gum	0.2	_
	Paraffinum liquidum	4	4
	Cetearyl alcohol	2	2
	PEG-20 stearate	0.5	0.5
	Cetyl alcohol	0.5	0.5
15	Petrolatum	1	1
	Theobromo cacao	1.5	1.5
	внт	0.02	0.02
	Dimethicone	5	5
	Tetrasodium EDTA	0.05	0.05
20	Gel of the invention	-	0.25
	Preservative	q.s	q.s
	Pigments	q.s	q.s

# Method:

# 25 Stage 1

EDTA was added to the water and dispersed with stirring. In sample 1, the Xanthan gum was added with stirring (pre-dispersed in glycerin and butylene glycol). In sample 2, the gel of the invention was added with stirring, followed by glycerin and butylene glycol. This phase was then heated to 70°C.

# 30 Stage 2

The oil phase was mixed in a separate vessel and heated to 70°C until melted and

uniform.

# Stage 3

The oil phase was added to the aqueous phase and an emulsion was formed using homogenisation for 10 minutes. Maintaining the shear, the pigments were slowly added and dispersed.

# Stage 4

5

The emulsion was then cooled to below 35°C with stirring. The preservative was added and the product was made to weight with purified water.

- Advantages of using a gel of the invention:
  - -Dispersing aid for the pigments
  - -Suspending the pigments
  - -Emulsion stabiliser
  - -Rheology modifier
- 15 -Improves textural properties
  - -Results in a conditioned end feel.

# Formulation Example 23 - Cream Foundation (1)

		Sample 1	Sample 2
20		%w/w	%w/w
	Aqua	69.4	69.2
	Stearic acid	20	20
	Lanolin	1	1
	Polyoxypropylene 15 stearyl ether	3	3
25	Glycerol monostearate (and) polyoxyethylene	3	3
	Stearate fatty acid ester		
	Propylene glycol	3	3
	Triethanolamine	0.6	0.6
	Gel of the invention	<del>-</del>	0.2
30	Pigments	q.s	q.s
	Preservative	q.s	q.s

# Stage 1

Into the water, propylene glycol (and gel of the invention in sample 2) was added with stirring. The remaining materials were added in sequential order (excluding the pigments and preservative).

This was then heated to 70°C with stirring.

Stage 2

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An emulsion was formed using high shear homogenisation for 10 minutes.

Maintaining the shear, the pigments were slowly added and dispersed. The emulsion was sheared until uniform.

Stage 3

The emulsion was then cooled to below 35°C with stirring. The preservative was added and the product was made to weight with purified water.

- Advantages of using a gel of the invention:
  - -Dispersing aid for the pigments
  - -Suspending the pigments
  - -Emulsion stabiliser
  - -Rheology modifier
- 20 -Improves textural properties
  - -Results in a conditioned end feel.

# Formulation Example 24 - Cream Foundation (2)

		Sample 1	Sample 2
25		%w/w	%w/w
	Aqua	73.7	73.7
	1,3-butylene glycol	8	8
	Cetearyl isononanoate	5	5
	Dimethicone	3	3
30	Glycerin	2	2
	Silica	1.5	1.5

	Caprylic/capric tryiglcerides	1.5	1.5
	Paraffinum liquidum	1.2	1.2
	Petrolatum	1.6	1.6
	Cetearyl octanoate	1	1
5	Cetearyl alcohol	1	1
	Talc	0.8	0.8
	Glyceryl stearate	0.8	0.8
	Lactic acid	0.4	0.4
	Xanthan gum	0.1	-
10	Tetrasodium EDTA	0.05	0.05
	Gel of the invention	-	0.3
	Pigments	q.s	q.s
	Preservative	q.s	q.s

20

#### Stage 1

EDTA and Lactic acid were added to the water and dispersed with stirring. In sample 1, the Xanthan gum was added with stirring (pre-dispersed in glycerin and butylene glycol). In sample 2, the gel of the invention was added with stirring, followed by glycerin and butylene glycol. This phase was then heated to 70°C.

# Stage 2

The oil phase was mixed in a separate vessel and heated to 70°C until melted and uniform.

#### Stage 3

The oil phase was added to the aqueous phase and an emulsion was formed using high shear homogenisation for 10 minutes. Maintaining the shear, the pigments (together with the talc and silica) were slowly added and dispersed. The emulsion was sheared until uniform. The emulsion was then cooled to below 35°C with stirring. The preservative was added and the product was made to weight with purified water.

Advantages of using a gel of the invention:

- -Dispersing aid for the pigments
- -Suspending the pigments
- -Emulsion stabiliser
- 5 -Rheology modifier
  - -Improves textural properties
  - -Results in a conditioned end feel.

# Formulation Example 25 - Face Powder/Granules (1)

10		Sample 1	Sample 2
		%w/w	%w/w
	Aqua	2.85	2.85
	Titanium Dioxide	23	23
	Mica	47	47
15	Talc	18	18
	Carboxymethyl cellulose	0.4	-
	Sorbitol	2	2
	Glyceryl monostearate	1.75	1.75
20	Lanolin	5	5
	Gel of the invention	-	0.4
	Pigments	q.s	q.s
	Preservative	q.s	q.s

#### Method:

# 25 Stage 1

Sorbitol was stirred into the water. Carboxymethyl cellulose (and gel of the invention in sample 2) was added to this with stirring. Lanolin and glyceryl stearate were added and this phase was heated to 70°C until melted and uniform.

#### Stage 2

Using stirring Titanium dioxide, Mica, Talc and pigments were added and dispersed.A Diosna mixer could be used to aid the dispersion.

Stage 3

The product was cooled to 35°C with stirring. The preservative was added and the product was made to weight with purified water.

- 5 Advantages of using a gel of the invention:
  - -Deposition
  - -Enhanced wear
  - -Enhanced structure
  - -Improves textural properties
- -Results in a conditioned end feel.

# Formulation Example 26 - Face Powder / Granules (2)

		Sample 1	Sample 2
		%w/w	%w/w
15	Aqua	3.2	3.2
	Talc	74	76
	Microcrystalline cellulose	10	10
	Magnesium stearate	6	6
	1,3-butylene glycol	6	6
20	Gel of the invention	-	0.8
	Pigments	q.s	q.s
	Preservative	q.s	q.s

#### Method:

# 25 Stage 1

Microcrystalline cellulose (and gel of the invention in sample 2) was dispersed in butylene glycol. Magnesium stearate was added and this phase was heated to  $70^{\circ}$ C with stirring.

# Stage 2

Using stirring, Talc and pigments were added and dispersed. A Diosna mixer could be used to aid this dispersion.

# Stage 3

The product was cooled to 35°C with stirring. The preservative was added and the product was made to weight with purified water.

- 5 Advantages of using a gel of the invention:
  - -Deposition
  - -Enhanced wear
  - -Enhanced structure
  - -Improved textural properties
- -Results in a conditioned end feel.

# Formulation Example 27 - Toothpaste

		Sample 1	Sample 2
		%w/w	%w/w
15	Aqua	47	47
	Sorbitol	30	30
	Hydrated silica	16	16
	Sodium lauryl sulfate	1.2	1.2
	Cellulose gum	0.85	-
20	Sodium monofluorophosphate	0.75	0.75
	Sodium saccharin	0.25	0.25
	Sodium hydroxide	0.2	0.2
	Gel of the invention	-	0.4
	Glycerin	3	3
25	Triclosan	0.3	0.3
	Flavour	q.s	q.s
	Preservative	q.s	q.s

#### Method:

# 30 Stage 1

The materials (excluding the flavour and preservative) were added to the water in

order, with stirring (Triclosan was pre-dispersed in glycerin). This was heated to 70°C maintaining stirring until uniform.

#### Stage 2

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A fryma mixer was then used to generate a uniform paste. This was mixed for 10 minutes. The product was then cooled to 35°C. The preservative and flavour were added and the product was made to weight with purified water. The product was mixed until uniform.

Advantages of using a gel of the invention:

- -Stable, reproducible viscosity build.
  - -Rheology modifier.
  - -Enhanced mouth feel.
  - -Stabiliser.
  - -Increased dwell time in the mouth.
- -Enhanced delivery of Triclosan.
  - -Improves textural properties

# Formulation Example 28 - Toothgel

		Sample 1	Sample 2
20		%w/w	%w/w
	Aqua	53	53
	Glycerin	45	45
	Carbomer	1	-
	PEG-40 hydrogenated castor oil	0.2	0.2
25	Sodium hydroxide	0.25	0.25
	Sodium fluoride	0.024	0.024
	Gel of the invention	-	0.5
	Triclosan	0.3	0.3
	Flavour	q.s	q.s
30	Preservative	q.s	q.s

Sample 1

Sample 2

#### Stage 1

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In sample 1, the Carbomer was added to the water and hydrated using homogenisation for 30 minutes. In sample 2, the gel of the invention was added to the water with stirring. The remaining materials (excluding the flavour and preservative) were added in order (the Triclosan was pre-dispersed in the glycerin). This was heated to 70°C maintaining stirring until uniform.

#### Stage 2

A fryma mixer was then used to generate a uniform paste. This was mixed for 10 minutes. The product was then cooled to 35°C. The preservative and flavour were added and the product was made to weight with purified water. The product was mixed until uniform.

Advantages of using a gel of the invention:

- -Stable, reproducible viscosity build.
  - -Rheology modifier.
  - -Enhanced mouth feel.
  - -Stabiliser.
  - -Increased dwell time in the mouth.
- 20 -Enhanced delivery of Triclosan.
  - -Improves textural properties

#### Formulation Example 29 - Body Scrub

		Sample 1	Sample 2
25		%w/w	%w/w
	Aqua	66	65.7
	Lauroamphodiacetate	8	8
	Sodium lauryl sulfate	6	6
	Hexylene glycol	6	6
30	PEG-40 castor oil	4	4
	Polysorbate 80	1.5	1.5

Cetyl acetate	1	1
Acetylated lanolin alcohol	0.5	0.5
Methyl gluceth-20	2	2
Glycerin	4	4
Polyethylene	1	1
Gel of the invention	-	0.3
Parfum	q.s	q.s
Preservative	q.s	q.s

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#### Stage 1

The materials (excluding the parfum, preservative and polyethylene) were added to the water in order with stirring, including gel of the invention in sample 2. This was heated to 70°C, maintaining the stirring. The bulk was stirred until uniform.

# 15 Stage 2

Maintaining the stirring the product was cooled to 35°C. The parfum, preservative and polyethylene were added and the product was made to weight with purified water. The product was stirred until the polyethylene was evenly distributed and the product was uniform.

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Advantages of using a gel of the invention:

- -Viscosity control
- -Rheology modifier
- -Skin conditioning effect
- 25 -Stabiliser
  - -Suspending the polyethylene beads
  - -Improves textural properties
  - -Results in a conditioned end feel.

# 30 Formulation Example 30 - Face Mask(1)

		%w/w	%w/w
	Aqua	80	84.5
	Kaolin	15	10
	Ethanol	5	5
5	Glycerin	5	5
	Gel of the invention	-	0.5
	Parfum	q.s	q.s
	Preservative	q.s	q.s

#### Stage 1

Kaolin, glycerin (and Gel of the invention in sample 2) were added to the water with stirring. This was heated to 70°C. A Torrence mixer was used to generate a uniform mix.

# 15 Stage 2

The bulk was cooled to 35°C and the remaining materials were added. The product was then made to weight with purified water and stirred until uniform.

Advantages of using a gel of the invention:

# 20 -Film-forming

- -Rheology modifier
- -Improves textural properties
- -Skin conditioning effect resulting in improved Skin feel
- -Results in a conditioned end feel

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# Formulation Example 31 - Face Mask (2)

		Sample 1	Sample 2
		%w/w	%w/w
	Aqua	65,75	66.15
30	Kaolin	12.5	12.5
	Glycerin	7	7

	Paraffinum liquidum	5	5
	Caprylic/capric triglyceride	2	2
	Talc	1.5	1.5
	Cetyl alcohol	2.25	2.25
5	Petrolatum	1.5	1.5
	Glyceryl stearate	1.5	1.5
	Cellulose gum	0.5	-
	Hydroxyethyl cellulose	0.5	-
	Gel of the invention	-	0.6
10	Preservative	q.s	q.s
	Parfum	q.s	q.s

#### Stage 1

The following materials were added to the water with stirring: Kaolin, glycerin, (hydroxyethyl cellulose and cellulose gum in sample 1 and gel of the invention in sample 2). This was heated to 75°C, maintaining the stirring. A torrence mixer was used to generate a uniform mix.

#### Stage 2

The oil phase, with the talc was mixed in a separate vessel and heated to 70°C until melted and uniform.

#### Stage 3

The oil phase was added to the aqueous phase and an emulsion was formed using 10 minutes homogenisation. The emulsion was then cooled to 35°C with stirring.

# 25 Stage 4

The preservative and perfume were added and the product was made to weight with purified water and stirred until uniform.

Advantages of using a gel of the invention:

# 30 -Film-forming

-Rheology modifier

- -Improves textural properties
- -Results in a conditioned end feel.

# Formulation Example 32 - Inorganic Sunscreen Formula (1)

5		Sample 1	Sample 2
		%w/w	%w/w
	Aqua	60	59.8
	Cetyl dimethicone copolyol	1.5	1.5
	Glyceryl oleate	2	2
10	Dimethicone	3	3
	Isopropyl palmitate	8	8
	Ceresin	1	1
	Paraffinum liquidum	4	4
	Dicaprylyl maleate	6	6
15	Titanium dioxide MT100T	5	5
	1,3-butylene glycol	8	8
	Magnesium sulphate	1	1
	Gel of the invention	-	0.2
	Preservative	q.s	q.s

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Method:

#### Stage 1

Magnesium sulphate, butylene glycol (and gel of the invention in sample 2) were added to the water using stirring. This phase was heated to 70°C maintaining stirring.

# 25 Stage 2

The oil phase was mixed and heated to 70°C until melted and uniform. At 70°C the titanium dioxide was added and dispersed using homogenisation.

#### Stage 3

Using stirring, the aqueous phase was slowly added to the oil phase and stirred until emulsified and uniform. The emulsion was then transferred to a homogeniser and high shear was applied for 5 minutes.

Stage 4

The emulsion was cooled to below 35°C with stirring and the preservative was added. Stirring continued until cool and uniform.

# 5 Advantages of using a gel of the invention:

- -Wetting agent for the titanium dioxide
- -Improves textural properties
- -Results in a conditioned end feel.

# 10 <u>Formulation Example 33</u> - Inorganic Sunscreen Formula (2)

		Sample 1	Sample 2
		%w/w	%w/w
	Aqua	52	51.8
	Cetyl dimethicone copolyol	1.5	1.5
15	Glyceryl oleate	2	2
	Dimethicone	3	3
	Isopropyl palmitate	8	8
	Ceresin	1	1
	Paraffinum liquidum	4	4
20	Dicaprylyl maleate	6	6
	Titanium dioxide MT100T	3	3
	Zinc oxide (Nanox)	10	10
	1,3-butylene glycol	8	8
	Magnesium sulphate	1	1
25	Gel of the invention	-	0.2
	Preservative	q.s	q.s

#### Method:

# Stage 1

Magnesium sulphate, butylene glycol (and gel of the invention in sample 2) were added to the water using stirring. This phase was heated to 70°C maintaining stirring.

# Stage 2

The oil phase was mixed and heated to 70°C until melted and uniform. At 70°C the titanium dioxide and zinc oxide were added and dispersed using homogenisation.

# Stage 3

Using stirring, the aqueous phase was slowly added to the oil phase and stirred until emulsified and uniform. The emulsion was then transferred to a homogeniser and high shear was applied for 5 minutes.

#### Stage 4

The emulsion was cooled to below 35°C with stirring and the preservative was added. Stirring continued until cool and uniform.

Advantages of using a gel of the invention:

- -Wetting agent for the titanium dioxide
- -Improves textural properties
- 15 -Results in a conditioned end feel.

# Formulation Example 34 - Pain Relief Syrup

		Sample 1	Sample 2
		%w/w	%w/w
20	Aqua	30	31
	Glycerin	30	30
	Sorbitol	35	35
	Microcrystalline cellulose	2	2
	Hydroxyethylcellulose	0.3	-
25	Acetaminophen	2	2
	Potassium acesulfame	0.05	0.05
	Gel of the invention	-	0.4
	Preservative	q.s	q.s
	Flavour	q.s	q.s
• •		1	

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Method:

#### Stage 1

The following materials were added to the water with stirring: Glycerin, sorbitol, microcrystalline cellulose (hydroxyethylcellulose in sample 1 and gel of the invention in sample 2). This was heated to 70°C with maintained stirring.

# 5 Stage 2

The bulk was then cooled to 35°C with stirring. Acetaminophen, potassium acesulfame, preservative and flavourings were then added and the product was made to weight with purified water. The stirring continued until the active was evenly distributed and the product was uniform.

Commis 1 Commis 2

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Advantages of using a gel of the invention:

- -Suspension of active
- -Viscosity control
- -Delivery of active
- 15 -Improves textural properties

#### Formulation Example 35 - Mouthwash

		Sample 1	Sample 2
		%w/w	%w/w
20	Aqua	81	81
	Ethanol	5	5
	Glycerin	8	8
	Sorbitol	5	5
	Sodium saccharin	0.025	0.025
25	Sodium fluoride	0.05	0.05
	Polysorbate 20	0.2	0.2
	PEG-40 hydrogenated castor oil	0.15	0.15
	Gel of the invention	-	0.1
	Preservative	q.s	q.s
30	Flavour	q.s	q.s

# Stage 1

Glycerin, sorbitol (and gel of the invention in sample 2) were added to the water with stirring. This was heated to 70°C, maintaining stirring.

# 5 Stage 2

The bulk was then cooled to 35°C. Sodium saccharin, sodium fluoride, polysorbate 20, PEG-40 and Ethanol were pre-mixed together and added to the bulk. The preservative and flavour were added and the product was made to weight with purified water. Stirring was maintained until the product was cool and uniform.

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Advantages of using a gel of the invention:

- -Enhanced mouth feel
- -Increased dwell time
- -Enhanced delivery of actives

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# Formulation Example 36 - Hair Gel

		Sample 1	Sample 2
		%w/w	%w/w
	Aqua	99	99
20	Carbomer 940	0.35	-
	Glycerin	0.5	0.5
	Tetrasodium EDTA	0.05	0.05
	Potassium Hydroxide	0.06	0.06
	Gel of the invention	-	0.35
25	Preservative	q.s	q.s

Method

Stage 1

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EDTA was dispersed in the water using stirring. For sample 1, the Carbomer was

then added and hydrated using homogenisation for 30 minutes. For sample 2, the gel was added and dispersed using stirring.

Glycerin was then added and mixed until uniform using stirring. Sample 2 was then heated to 70°C (sample 1 remained at room temperature).

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# Stage 2

Sample 2 was cooled to below 35°C using stirring. The preservative was then added to both samples and they were made to weight with purified water. The gels were stirred until cool and uniform.

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# Advantages of using a gel of the invention

- -Stable, reproducible viscosity build.
- -Rheology modifier.
- 15 -Hair conditioning effect.
  - -Hair styling and hold
  - -Improved textural properties.
  - -Results in a conditioned end feel.

# 20 <u>Formulation Example 37</u> - Sun Lotion

		Sample 1	Sample 2
		%w/w	%w/w
	Aqua	64.8	59.45
	Ethanol	10	10
25	Octyldodecanol	8	8
	Glycerin	5	5
	Octyl methoxycinnamate	-	2
	Octocrylene	-	1
	Hydrogenated coco-glycerides	3	3
30	Butyl methoxydibenzoylmethane	-	2
	Drometrizole trisiloxane (Mexoryl XL)	3	3

	Terephthalylidene dicamphor sulfonic acid (Mexoryl SX)	3	3
	4-methylbenzylidene camphor	1	1
	C12-15 alcohols benzoate	1	1
	Coco-glucoside	1	1
5	Theobromo cacao	0.5	0.5
	Tocopheryl acetate	0.2	0.2
	Gel of the invention	0.35	0.35
	Preservative	q.s	q.s
	Parfum	q.s	q.s

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Method

Stage 1

EDTA and glycerin were dispersed in the water using stirring. Gel of the invention was then added and dispersed using stirring. This phase was then heated to 70°C.

# Stage 2

The oil phase was mixed, including the sunscreens and heated to 70°C.

# Stage 3

With the temperature of both phases at 70°C, the oil phase was added to the aqueous phase and an emulsion was formed using high shear homogenisation for 10 minutes

# Stage 4

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The emulsion was cooled to below 30°C with stirring. Tocopheryl acetate, Ethanol, preservatives and perfume were all added with stirring. The emulsion was made to weight with purified water and cooled to room temperature.

Advantages of a gel of the present invention

- -Stable, reproducible viscosity build.
- -Rheology modifier.
- -Enhanced SPF efficacy.
- 5 -Improved textural properties.

# Formulation Example 38 - Sun Spray Lotion

		Sample 1	Sample 2
10		%w/w	%w/w
	Aqua	64.3	59.45
	Ethanol	10	10
	Octyldodecanol	8	8
	Glycerin	5	5
15	Octyl methoxycinnamate	_	2
	Octocrylene	-	1
	Hydrogenated coco-glycerides	3	3
	Butyl methoxydibenzoylmethane	-	2
	Drometrizole trisiloxane (Mexoryl XL)	3	3
20	Terephthalylidene dicamphor sulfonic acid (Mexoryl SX)	3	3
	4-methylbenzylidene camphor	1	1
	C12-15 alcohols benzoate	1	1
	Coco-glucoside	1	1
	Tocopheryl acetate	0.2	0.2
25	Gel of the invention	0.35	0.35
	Tetrasodium EDTA	0.05	0.05
	Preservative	q.s	q.s
	Parfum	q.s	q.s

Stage	1

EDTA and glycerin were dispersed in the water using stirring. Gel of the invention was then added and dispersed using stirring. This phase was then heated to 70°C.

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Stage 2

The oil phase was mixed, including the sunscreens and heated to 70°C.

10 Stage 3

With the temperature of both phases at 70°C, the oil phase was added to the aqueous phase and an emulsion was formed using high shear homogenisation for 10 minutes

15 Stage 4

The emulsion was cooled to below 30°C with stirring. Tocopheryl acetate, Ethanol, preservatives and perfume were all added with stirring. The emulsion was made to weight with purified water and cooled to room temperature.

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Advantages of a gel of the present invention

- -Stable, reproducible viscosity build.
- -Rheology modifier.
- 25 -Enhanced SPF efficacy.
  - -Improved textural properties.

Formulation Example 39 - Sustained Release Zinc and Vitamin C Tablets

Sample 1 Sample 2

%w/w

%w/w

	Ascorbic acid	66	66
	Xanthan gum	10	-
	Gel of the invention	-	10
	Sorbitol	17.5	17.5
5	Magnesium stearate	1	1
	Zinc oxide	5.5	5.5

# 10 Stage 1

The powdered components of the gel of the invention were added to water, heated to over 65°C and then cooled with stirring to 35°C.

# 15 Stage 2

The ingredients were passed through a 30 mesh sieve in order, blended together in a turbula and then compressed to form the tablet.

# Advantages of a gel of the present invention

- -Sustained release of actives
- -Enhances delivery of actives
- -Improved tablet structure

#### **CLAIMS**

- 1. A fluid gel obtainable by a process which comprises subjecting a solution comprising a mixture of xanthan and one or more non-gelling polysaccharides selected from galactomannans and glucomannans in a solvent to shear while cooling the solution from a temperature above the gelation temperature of the mixture, and recovering the resulting continuous gel.
- 2. A gel according to claim 1 wherein the solution comprises a mixture of xanthan and konjac mannan.
- 3. A gel according to claim 1 wherein the solution comprises a mixture of xanthan and locust bean gum.
- 4. A gel according to any one of claims 1 to 3 wherein the xanthan and one said non-gelling polysaccharide are present in a weight ratio of from 1:10 to 10:1.
- 5. A composition comprising a gel as defined in any one of claims 1 to 4 and a physiologically tolerable carrier, adjuvant or diluent.
- 6. A composition according to claim 5 which further comprises one or more of a pigment, a pharmaceutically active compound, a sunscreen active component, a silicone or derivative thereof, an inorganic salt or a solvent.
- 7. A composition according to claim 6 wherein one or more of the further components is added to the solution before subjecting the solution to shearing.
- 8. A composition according to claim 5, 6 or 7 which takes the form of a topical, oral or mucosal contact gel or lotion, or a viscous fluid.
- 9. A process for producing a fluid gel, which process comprises subjecting a solution comprising a mixture of xanthan and one or more non-gelling polysaccharides selected from galactomannans and glucomannans in a solvent to shear while cooling the solution from a temperature above the gelation temperature of the mixture, and recovering the resulting continuous gel.
- 10. A process according to claim 9 which further comprises the preliminary step of heating the said mixture to a temperature above the gelation

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temperature of the mixture.

- 11. A process according to claim 9 or 10 wherein the solution comprises 0.05 to 2% by weight of the mixture of xanthan and one or more non-gelling polysaccharides.
- 5 12. A process according to any one of claims 9 to 11 wherein the solvent is chosen from water, alcohols, ketones and mixtures thereof.
  - 13. A process according to any one of claims 9 to 12 wherein the solution before cooling is at a temperature from just above the gelation temperature of the mixture to 95°C.
  - 14. A process according to any one of claims 9 to 13 wherein the solution is cooled to the gelation temperature of the mixture or below.
    - 15. A process according to any one of claims 9 to 14 wherein the solution is cooled to room temperature.
  - 16. A process according to any one of claims 9 to 15 which further comprises, in either order, (i) heating the xanthan and the said one or more non-gelling polysaccharides to a temperature greater than or equal to the gelation temperature of the mixture and (ii) mixing the xanthan and the said one or more non-gelling polysaccharides together.
  - 17. A cosmetic or pharmaceutical composition obtainable by a process which comprises:
    - (a) forming an aqueous phase comprising one or more cosmetically or pharmaceutically acceptable carriers, adjuvants or diluents and a mixture of xanthan and one or more non-gelling polysaccharides selected from galactomannans and glucomannans;
    - (b) Subjecting the aqueous phase to shear while cooling it from a temperature above the gelation temperature of the said mixture; and
    - (c) recovering the resulting cosmetic or pharmaceutical composition.
  - 18. A process for producing a cosmetic or pharmaceutical composition which process comprises:
    - (a) forming an aqueous phase comprising one or more cosmetically or pharmaceutically acceptable carriers, adjuvants or diluents and a

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- mixture of xanthan and one or more non-gelling polysaccharides selected from galactomannans and glucomannans;
- (b) Subjecting the aqueous phase to shear while cooling it from a temperature above the gelation temperature of the said mixture; and
- (c) recovering the resulting cosmetic or pharmaceutical composition.
- 19. A fluid gel whenever obtained by a process as defined in any one of claims 9 to 16.
- 20. A gel as defined in any one of claims 1 to 4 for use in a method of diagnosis practised on the human or animal body.
- 21. A gel as claimed in claim 20 wherein the method of diagnosis comprises an ultrasound technique.

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- 22. A gel according to any one of claims 1 to 4 and specifically hereinbefore mentioned.
- 23. A composition according to any one of claims 5 to 8 and 17 and substantially as hereinbefore described.
  - 24. A process as defined in any one of claims 9 to 16 and 18 and substantially as hereinbefore described in any one of the examples.







Application No:

GB 0014865.0

Claims searched: 1-24

Examiner: Date of search:

Martin Price 17 August 2001

# Patents Act 1977 Search Report under Section 17

# Databases searched:

UK Patent Office collections, including GB, EP, WO & US patent specifications, in:

UK Cl (Ed.S): C3U - UBD, UFB

Int Cl (Ed.7): C08J 3/075; C08L 5/00

Other: Online - WPI, EPODOC, JAPIO

# Documents considered to be relevant:

Category	Identity of document and relevant passage		Relevant to claims
Y	GB 2310668 A	(J+J) -see eg page 4 line 35 and example 1	1 at least
Y	EP 0355908 A1	(Unilever) - see eg claims 8-10	1 at least
Y	EP 0208313 A2	(Morinaga) - see the claims and examples	1 at least
Y	US 4676976	(Ajinomoto) - see eg claim 4	1 at least
Y	US 4647470	(Merck) - see eg examples 11-13	1 at least
Y	WPI Accession number 1987-076427[11] & JP 62029947 (Ajinomoto) - see abstract		1 at least
Y	WPI Accession num	nber 1985-118772[20] & JP 60058443 (Kohjin) - see	1 at least

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- A Document indicating technological background and/or state of the art.
- P Document published on or after the declared priority date but before the filing date of this invention.
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